2006

Developing a quality control program for an NIRS -based service and research laboratory

Maureen Suryaatmadja
Iowa State University

Developing a quality control program for an NIRS –based service and research laboratory

by

Maureen Suryaatmadja

A thesis submitted to the graduate faculty

in partial fulfillment of the requirements of the degree of

MASTER OF SCIENCE

Major: Agricultural Engineering (Food and Process Engineering)

Program of Study Committee:
Charles R. Hurburgh, Jr, Major Professor
Carl J. Bern
T.B. Bailey

Iowa State University

Ames, Iowa

2006

UMI Number: 1439910

UMI Microform 1439910

ProQuest Information and Learning Company
300 North Zeeb Road
P.O. Box 1346
Ann Arbor, MI 48106-1346

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACKNOWLEDGEMENTS

I would like to thank Dr. Charles Hurburgh for the opportunity to work in Grain Quality Lab and all helps that you have provided. I also want to thank Dr. Carl Bern and Dr. Ted Bailey for serving as the program of study committee for my research. The supervision and guidance provided by the committee has been incredibly valuable in accomplishing my research goals. I also would like to thank Glen Rippke for helping me to be familiar with lab activities. In addition, I would like to thank Robert and Igor for your helps. Finally, I would like to thank my parents, my brother, my boyfriend, and my friends for their understanding, patience and support while I conducted research and wrote my thesis. Your supports have been much appreciated.

ABSTRACT

The Iowa State University Grain Quality Laboratory (ISU-GQL) provides instrument calibration and measurement services for composition of agricultural products. The Laboratory is pursuing ISO 17025 accreditation. Quality control is one of the ISO 17025 requirements, therefore the objectives of this study are to develop quality control program for Near Infrared Spectroscopy (NIRS), supporting equipment and laboratory room conditions and to develop quality control program for the reference data. The first quality control program includes data from NIRS daily check samples, NIRS duplicate differences, NIRS real time prediction comparison to references, periodic checks and reviews of equipment supporting the NIRS calibration, and a record of laboratory climate conditions. The second quality control program consists of internal data (oven moisture and corn density) which are done by ISU-GQL and external data (proximate analysis, amino acids and fatty acids) which are done by other labs. The quality control activities include setting tolerances, developing appropriate control charts, handling and documenting data, writing Standard Operating Procedures (SOP) of quality control activities, implementing quality control program and estimating cost of quality. The tolerance setting of NIRS that was based on the Standard Error of Prediction (SEP) as described in AACC Method 39-00 appears to give a better control than shewhart control chart. The quality controls for supporting equipment improved the consistency of data generated by the lab. Climate data provides basis to look at error pattern of other data. A better documentation of reference data that utilized Access database gives benefit of easier data searching. By establishing quality control program, ISU-GQL satisfies some objectives of quality control and ISO 17025 requirements.

CHAPTER 1. GENERAL INTRODUCTION AND LITERATURE REVIEW

Analytical laboratories are essential organizations in numerous industries, academic institutions and regulatory agencies. Laboratories can perform their services in many fields such as chemistry, microbiology, nutrition, food science, pharmaceutical and agriculture. In all these areas it is necessary for the laboratory to generate repeatable data that meets client needs and satisfies the required or implied quality standard (Garfield et. al., 2000). In addition, analytical laboratories pursuing accreditation must meet the requirements stated in ISO (International Standard Organization) 17025. ISO 17025 is a general requirement for competence of testing and calibration laboratories (Tholen, 2002).

The Iowa State University Grain Quality Laboratory (ISU-GQL) is an analytical laboratories pursuing accreditation. ISU-GQL performs calibration activity for Near Infrared Spectroscopy (NIRS) units and other analytical technologies. ISU- GQL also provides services for measuring the chemical composition and biological properties of agricultural products (primarily corn and soybean) using NIRS instruments.

There are two principal factors that determine NIRS performance. The first factor is a spectrum, which is generated from the interaction of the measuring instrument with the unique optical and chemical characteristics of a sample. The second factor is the reference value(s) of the samples used for calibration. Reference values are those chemical or physical parameters to be predicted using spectroscopic measurements (Workman, 1996).

Figure 1 explains the NIRS calibration process as done in ISU-GQL. The calibration process is begun with optical scanning of samples in NIRS instruments (about 400-

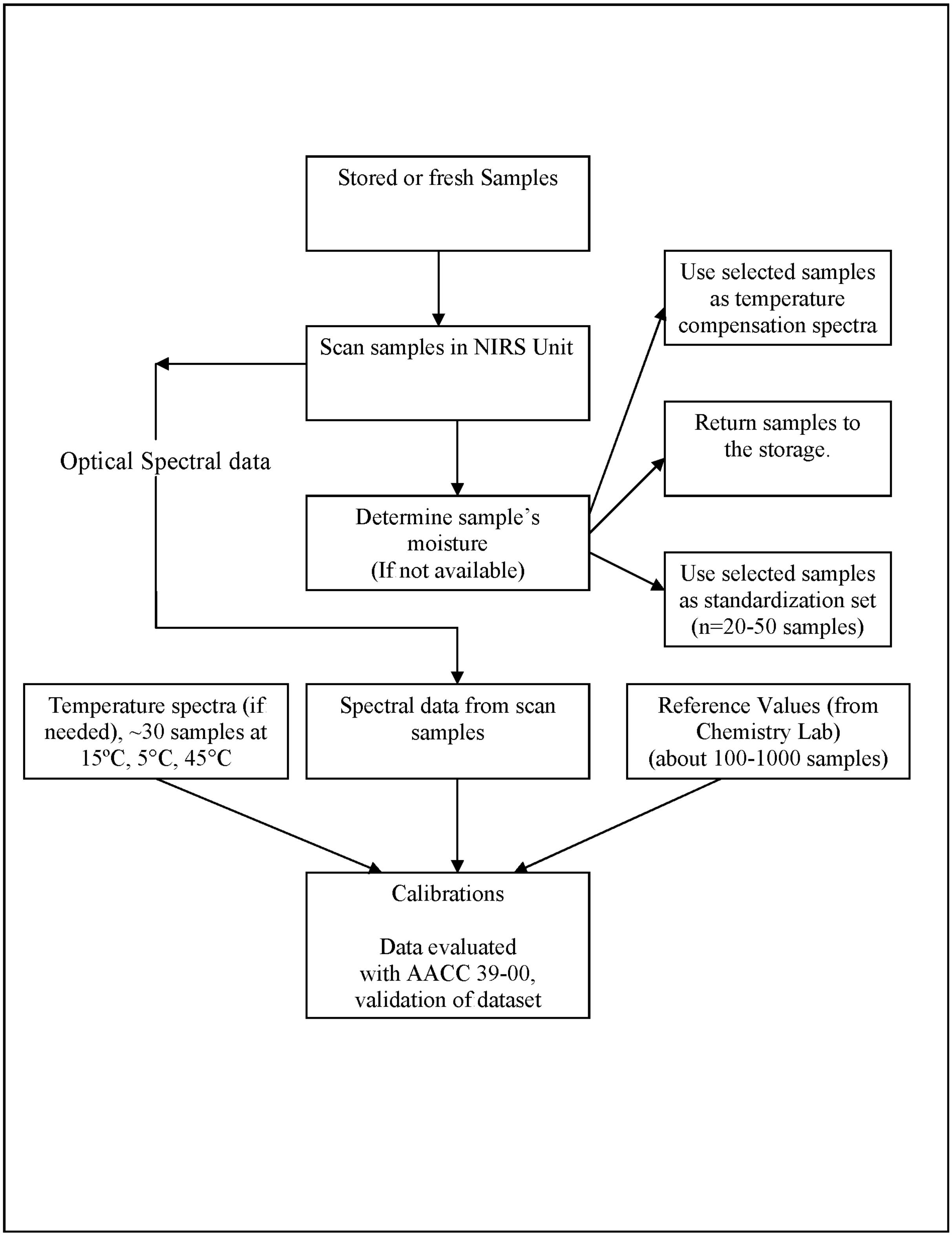


Figure 1. NIRS Calibration Flowchart at ISU-GQL

1000 samples per year). The sample moisture is measured immediately using an oven moisture method or another indirect method if the sample must be preserved. Twenty to fifty samples are used for standardization sample set and the rest are sent back to the storage. The chosen samples (about 100-1000 samples) are sent to the chemistry lab to get the reference data (if not already available). If temperature stabilization is needed, 30-40 samples are scanned at 15°C, 5°C, and 45°C. The calibration results are evaluated according to AACC Method 39-00 (AACC International 10^{th} Ed, 2000).

Accurate calibration of NIRS instruments means that the NIRS instruments, the reference values, all supporting equipment and the laboratory conditions must meet the required quality standard. The sources of error in NIRS testing can be divided into factors associated with the instruments, factors associated with the samples, and factors associated with the operators. The instrument factors include wavelength scale, photometric scale, instrument temperature control, cell covers, relative humidity, instrument to instrument variability and mathematical signal processing of the log (1/R) signal. The sample factors include sample chemical composition, bulk density, physical texture, temperature, ambient temperature, conversion factors, genetics and whole grain application. The operational factors include calibration practice, sample preparation, storage, ambient conditions and cell loading. The most troublesome sources of error are likely to be instrument-to-instrument variability with respect to wavelength, sample selection for calibration, sampling and sample preparation, wavelength selection and reference laboratory analysis (Williams and Norris, 2001a).

Instruments consist of mechanical, optical, or electronic components. Their performance can change as components age or wear. It is difficult to detect slow changes in day to day or even week to week operation, and, since most measurements are made by comparing the response of the sample with the response of the instrument, slow changes tend to be overlooked. If these subtle changes are not monitored, inaccuracy will eventually occur. As general rule, instrumental errors of this kind are controlled though a consistent practice of periodic calibration and validation (Garfield, 1991).

The supporting equipment is a critical element in the instrumental laboratory like ISU-GQL. There are three general principles in considering the quality assurance aspects of instruments and equipment: capability, condition/maintenance and monitoring. First, the equipment should be capable, by prior documentation, of making the test required. Second, all equipment should be kept in optimal condition for use. This implies both preventive maintenance and control over the use of the equipment by trained laboratory personnel. Third, equipment should be frequently monitored and evaluated (Dux, 1990). ISO 17025 section 5.5 requires laboratories to have equipment capable of producing results that meet the stated quality objectives. This includes relayed items or sampling, preparation of test and/or calibration items, processing and analysis of test and/or calibration (AOAC Training Course Series, 2002a).

Operators play an important role in generating data that meet quality standard. Operators have to be trained to operate instruments and equipment in the lab. In addition, operators must have a basic understanding of the role of the laboratory and some of the basic elements of the job itself (Garfield et al, 2000). Therefore, it is essential that new employees receive some form of orientation and training. ISO 17025 section 5.2 states that personnel performing specifics tasks are qualified on the basis of appropriate education, training, experiences and/or demonstrated skills, as required (AOAC Training Course Series International, 2002b).

The performance of measuring instruments and related equipment may be affected by environmental conditions. For example, uneven room temperature throughout the day in a laboratory will severely affect the retention times of room-temperature chromatographic procedures (Garfield, 1991). ISO 17025 section 5.3 requires that laboratory facilities for testing and/or calibration, including but not limited to energy sources, lighting and environmental conditions, shall be such as to facilitate correct performance of the tests and/or calibrations. The laboratory shall ensure that the environmental conditions do not invalidate the results or adversely affect the required quality of any measurement. The technical requirements for accommodation and environmental conditions that can affect the tests and calibrations shall be documented (AOAC Training Course Series, 2002c).

The ambient temperature can affect the performance of NIRS instruments, since detectors and other components are sensitive to temperature. This is particularly applicable to computerized spectrometers, which may not be equipped with the same degree of temperature control as commercial bench model instrument. If variations in ambient temperature are expected under conditions of future operation of the instrument, similar fluctuations should be introduced during period when the calibration is being developed. Each instrument and calibration should have these limits specified (Williams and Norris, 2001b).

Air humidity can affect samples that are opened in the laboratory. Samples can either gain or lose moisture depending upon the atmospheric conditions (Garfield, 1991). Changes in relative humidity also may change the noise level of a NIR spectrophotometer, particularly in the areas of maximum absorption by water bonds. This is especially true for instruments that use an empty cell (air) reference before test. Thus, the room condition needs to be either controlled or monitored (Williams and Norris, 2001c).

The consistency of reference data from the chemistry lab is essential in developing NIRS calibrations. The ISU-GQL obtains a reference chemistry values such as protein, oil, fiber, starch, amino acids and fatty acids from external laboratories. However, ISU-GQL does some types of reference values itself (oven moisture and corn density). In order to maintain consistency, both internal and external reference chemistry data need to be controlled. The reference chemistry values also need to be checked periodically. Reference checks are performed to verify, and qualify new data for calibration purposes (Hansen, 2004).

The reference data must be verified against itself over time which is usually called internal quality control. The simplest and cheapest internal quality control is a control chart. Providers of reference data should also participate in proficiency testing. Proficiency testing is a form of external quality control involving many laboratories measuring test portions of the same material. Proficiency testing provides assurance to laboratory management, accreditation bodies and customers that the results produced by laboratory are consistent with those produced by other laboratories. In cases where a reliable assigned value is available for the test material; good performance in the proficiency test gives assurance of the trueness (Mullins, 2003). Professional society, such us AOCS, provides organized proficiency test programs for some traits. The program objective is to achieve and maintain peak performance of laboratory staff and equipment. It is designed to fit in with accreditation needs under ISO 17025 (AOCS International, www.aocs.org, 2006).

Developing a quality control program for internal lab data is more straight forward than developing the quality control program for external lab data. The measurements in the lab can be monitored more regularly at lower cost. And, when the errors appear, the source of error can be directly searched. In the case of outsourced reference chemistry data, the errors may not be easily found or the source may not wish to provide internal tracking information, Outsourcing holds definite drawbacks, including potential loss of control, confidentiality and quick turnaround times. When a firm submits samples to a contract lab, it loses control over when the sample is done, who performs the work, and how testing is performed (Marsilli, 1997). Therefore, ISU- GQL must have assurance that the results of each reference submission can be accepted within a certain confidence level.

The ISU-GQL had collected reference chemistry reproducibility data for many years. However, the documentation of the data was poorly organized. Data documentation plays an important role in developing quality control. Poorly organized data will cause difficulties in analyzing and interpreting the data for future use.

The maintenance of records of analyses is as essential to a laboratory's operations as the various steps in the collection, analysis and storage of samples. The records have potential long term value and may serve many purposes. Sloppy records and poor records maintenance likely reflect poor quality control in other areas of operations and will give that impression to others (Garfield, 1991). ISO 17025 section 4.12 also states that the laboratory shall retain records of original observation, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each test report or calibration, for a defined period. The records for each test or calibration shall contain sufficient information to facilitate, if possible, identification of factors affecting the uncertainty and to enable the test or calibration to be repeated under conditions as close as possible to the original (AOAC Training Course Series, 2002d).

It is clear that quality control is an essential part of the ISO 17025 standard and the laboratory quality management system philosophy. Quality control is required in ISO 17025 and it is specifically stated in section 5.9. However, there is no requirement that quality be high, although this would be favorable for laboratories who want to keep and gain clients, only that quality be maintained at a level that is efficient for the laboratory (Tholen, 2002).

The purposes of establishing quality control program in a laboratory can be one, some or all of the following:

- to upgrade the overall quality of laboratory performance
- to maintain a continuing assessment of the quality of data generated by analysts
- to identify good analytical methods and research needs
- to address quality documentation requirements in research laboratories
- to provide a permanent record of instrument performance as basis for validating data and projecting repairs and replacement needs
- to ensure sample integrity
- to improve record keeping
- to produce analytical results that can withstand legal scrutiny
- to identify training needs
- to identify and eliminate sources of error

(Garfield et al, 2000).

The quality control operations within a laboratory make up a very important part of the quality assurance system. Compilation of statistical data, interpretation of the data, location of potential problems, and quantitative demonstration of performance make up the quality control operations. Quality control operations in an analytical laboratory may consist of:

- Instrument calibration
- Instrument maintenance
- Control Charting and Trend Analysis
- Proficiency Samples and Reference Samples
- Method Validation Studies

(Garfield et al, 2000).

In addition, other activities intended to control external influences on accuracy also can be included on the quality control operations.

Quality control always includes statistical process control. A useful tool in statistical process control is control charting. Control charts provide the means for displaying the quality control of a given analytical process. Control charts are strongly suggested when a sample with a known standard deviation and mean is run on a routine basis. A control chart based on mean has control limits which are used as criteria for action or for judging whether a set of data does or does not fall within control limits. The warning limits usually correspond to ±2 standard deviations from the mean. By using this limit, there is a 5% chance that a result will exceed the control limits. Action limits are normally set at ±3 standard deviations from the mean, in which case, there is only 0.3% chance that a result will exceed the control limits by random chance alone. The mean and standard deviation are calculated using following formula:

$$\text{Mean} = \bar{x} = \frac{\sum X_n}{n} \qquad \text{(eq.1)}$$

$$\text{standard deviation} = s = \sqrt{\frac{\sum (x-\bar{x})^2}{n-1}} \qquad \text{(eq.2)}$$

(Garfield et al, 2000).

The quality control system must be implemented and maintained in an efficient and effective manner that requires timely responses to problems. Key components are hardware design and implementation, training, system management, the collection of data to monitor performance, system updates and documentation (Morris, 1991).

Quality is always associated with costs. Quality has an impact on the costs of goods and services in an organization. The reasons to identify and measure the costs associated with poor quality are to quantify the size of the quality problem to help justify an improvement effort, to guide the development of the effort and to track progress in improvement activities. By evaluating quality and cost, the value of product/service and process output will be increase, and customer will be satisfied (Gryna, 1999).

The quality control programs as part of laboratory quality management system will give benefits once they are well planned and implemented. According to Westgard and Barry (1986), improved quality can lead to reduce costs because of the amount of money that is already being wasted by producing goods and service of unsatisfactory quality. Hurburgh (2003) also stated that organizations pursuing QMS can experience company growth, methods for problem recognition and resolution, time and effort savings, improvement in record maintenance, and overall laboratory consistency. When the system is fully implemented, it should be capable of outside third party audit/certification. Quality control programs, should reduce the cost of producing unsatisfactory quality service.

Previous study in ISU-GQL by Hansen (2004) had developed a quality management system to apply ISO 17025 accreditation. The work included writing and implementing laboratory procedures, standardizing job description, creating a quality manual, and generating working control charts. Although some control charts have been developed as part of quality control, they still needed improvement. The quality control program still needs to be created to meet ISO 17025 requirements.

The efficient and effective quality control program for NIRS analyzer, supporting equipment and environmental condition needs to be established in ISU-GQL. In addition, a quality control program for the reference data also should be developed in ISU-GQL.

CHAPTER 2. OBJECTIVES AND THESIS ORGANIZATION

The objectives of this research were

1) to develop an internal quality control program for the NIRS analyzers, supporting equipment and laboratory room conditions, and

2) to develop quality control program for the reference data, most of which is externally provided.

The essential elements of the internal program were

- NIRS daily check sample (control chart).
- Online NIRS Instrument Duplicates (control chart).
- Original NIRS values vs. Reference chemistry (control chart).
- Periodic check and reviews of other instruments supporting the NIRS calibration.
- Recording of laboratory room conditions (temperature, humidity, pressure) as a research tool for potential use in accuracy improvement.

Quality Control Program for fundamental/reference methods consisted of

- Replicates of measurements done by ISU GQL: oven moisture and corn density.
- Long term replication of reference data from external chemistry laboratories: Proximate analysis, Amino Acids and Fatty Acids.

The work included setting tolerances, developing appropriate control charts, handling and documenting data, implementing quality control program, writing procedures for quality control activities and estimating the cost of quality. Microsoft Word™ (Microsoft Corp, www.microsoft.com) and Microsoft Excel™ (Microsoft Corp, www.microsoft.com) along with custom macro-program routines were utilized to support quality control program in

ISU-GQL. Microsoft Access™ (Microsoft Corp, www.microsoft.com) were also introduced to handle the large amount of reference data.

Documentation of control charts were included to provide direction for future researchers. The procedures of quality control activities quality control operation, evaluation and monitoring, corrective action, data handling and documentation, and use of the data were also developed.

The thesis is written in chapter format. The study of developing quality control program for NIRS instruments, supporting equipment, laboratory room conditions and reference data will be submitted to the journal of Cereal Chemistry, published by American Association of Cereal Chemist (AACC).

CHAPTER 3. QUALITY CONTROL FOR NIRS ANALYZERS

Before designing a quality control program, for NIRS analyzers, basic NIRS theory, calibration, measurement and validation must be understood. The basic theory of NIRS was described by Hruschka (2001). When near-infrared radiation (700-2500nm) is incident on a solid sample, some of it is reflected from the surface of the sample, some is absorbed and some is transmitted through the sample. Figures 2 shows how the transmittance instrument work. The incident ray, E_I (known), will be transmitted in the transmittance instrument. The transmission instrument will measure the fraction transmitted (E_T). The amount of radiation transmitted from sample is quantified as the transmittance (R). The value is usually expressed as log (1/R) or log(1/A), which gives higher values at higher levels of absorbance. There is an almost linear relationship between log (1/R) and the concentration of an absorbing component. Bouger-Lambert-Beer Law (Beer's Law) described their relationship below:

$$\text{Log}\ (1/R_\lambda) = \alpha_\lambda\, L\, C \qquad \text{(eq.3)}$$

$\text{Log}\ (1/R_\lambda)$ = absorbance.

α_λ = molar absorption coefficient.

L = path length (thickness).

C = analyte concentration (reference data).

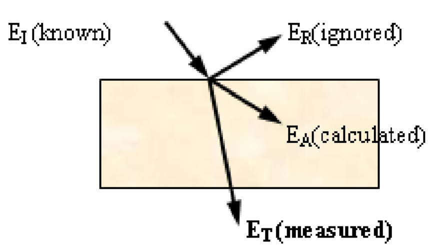


Figure 2. Transmittance Instruments

Log (1/R) is related to the amount of the component as determined by the reference method. Establishing this relationship using a set of samples of known composition is called calibration. The most common calibration is a multiple linear regression:

$$Y= a+b_1x_1+b_2x_2+\ldots+b_kx_k \qquad \text{(eq.4)}$$

x = log (1/R) values at k different wavelengths

The accuracy of a regression for a set of calibration samples is reported as the standard error of calibration (SEC), the correlation coefficient (r), or the coefficient of determination (r^2) (Hruschka, 2001).

Once an NIRS instrument has been calibrated against a reference method, it can be used to determine the percentage of a constituent in new samples. Theoretically, this measurement should have a measurement error roughly equal to the SEC. However, the NIRS measurement error may, in practice, be significantly larger than the SEC, if the new samples are not completely represented in the calibration set. Comparison of NIRS measurements and reference method measurements on a new set of samples provides a basis for calculation of the true measurement error. This comparison is called validation of the calibration. The mean ($\overline{D}$) and standard deviation (s_D) of the difference between the NIRS measured and reference values for several samples (validation/prediction set) estimate the systematic and random errors, respectively, of the NIRS method. The estimate $\overline{D}$ is sometimes called Bias and s_D is called Standard Error of Prediction (SEP). SEP is calculated using formula below:

$$SEP = \sqrt{\frac{\sum (D-\overline{D})^2}{(n-1)}} \qquad \text{(eq. 5)}$$

n= number of validation samples

(Hruschka, 2001).

NCWM Publication 14, Near Infrared Grain Analyzer states that SEP measures accuracy of the instrument with respect to the reference method (NCWM 14, 2004).

AACC Method 39-00 suggested that standard error of precision ≤ 0.33*SEP. (AACC Method). In other words, precision of single measurement could be estimated with a percentage of the accuracy (SEP). Precision expresses the closeness of agreement between repeated test results (Kateman and Buydens, 1993). On the assumption that individual test results follow a Normal Distribution with the standard deviation estimated by 0.33 SEP, then the difference between pairs will follow a Normal Distribution with mean is zero and standard error of difference (SED) is equal to $\sqrt{2}$ *0.33 SEP (Mullins, 2003).

The concept above then was used to set the tolerance for each quality control activity done for NIRS instruments in ISU-GQL. By using AACC Method 39-00, the same brand of instrument would have the same tolerance which means that they have the same accuracy and precision. But the tolerance of different NIRS instrument brands would be different because of different SEP. This method will be compared to the shewhart control chart method. A shewhart control chart based on mean has the warning limits that usually correspond to 95% confidence level or ±2 standard deviations from the mean. By using this limit, there is a 5% chance that a result will exceed the control limits. Mean and standard deviation are calculated using equation 1 and 2 in Chapter 1, on the control data itself, not as a prior estimate as the SEP calculation would be.

ISU-GQL calculated the accuracy based on cumulative average of SECV (Standard Error of Cross Validation), and SEP from some activities as described in table 1. These values were weighted equally because they are basically are the same process, but the samples used are different. When validation process uses part of calibration sample set, the standard error is called SECV. When the validation process uses sample set that are totally different from calibration sample set, the standard error is called SEP. The accuracy, precision and standard error of differences were calculated based on the 95% confidence level as suggested by AACC Method 78-60 (AACC International 10th Ed, 2000) in determining significance level. The cumulative average of accuracy, precision and standard error of difference was multiplied by two, since the 95% confidence level equals two standard deviations. The example of tolerance calculation for an Infratec NIRS instrument (corn protein) is shown in table 1. The other calculations can be seen in Appendix A. Tables 2,3,4,5, 6 and 7 show the summary of tolerances for each brand of instrument (corn and soybeans). The tolerance values are implemented in control charts which will be explained in the next two chapters.

Table 1. Infratec Corn Protein Tolerance Calculation

Item [a]	**CAL ID**	**SEP**	**Type**	**Cum. Ave**	**95% CI**	**0.33* SEP**	**95% CI**	**SED**	**95% CI**
Base calibration	CN200205	0.57	SECV	0.57	1.14	0.19	0.38	0.27	0.53
Base Calibration Temp+ Stabilization	CN200205	0.55	SECV	0.56	1.12	0.18	0.37	0.26	0.52
Original Calibration 2004	CN20030X	0.47	SECV	0.53	1.06	0.18	0.35	0.25	0.49
Base Calibration Temp+ Stabilization (Val 2004)	CN20030X	0.32	SECV	0.48	0.95	0.16	0.31	0.22	0.44
Validation 553075 (Crop 2004)	CN20030X	0.38	SEP	0.46	0.91	0.15	0.30	0.21	0.43
Validation 1241350 (Crop 2004)	CN20030X	0.48	SEP	**0.46**	**0.92**	**0.15**	**0.30**	**0.22**	**0.43**

[a] Basis 15% moisture

Table 2. Summary of Infratec Corn Tolerance Calculation

Constituent	SEP	95% CI	0.33* SEP	95% CI	SED	95% CI SED
Moisture (%)	0.55	1.10	0.18	0.36	0.26	0.51
Protein [a] (%)	0.46	0.92	0.15	0.30	0.22	0.43
Oil [a] (%)	0.40	0.80	0.13	0.26	0.19	0.37
Starch [a] (%)	0.78	1.56	0.26	0.52	0.36	0.73
Density [a] (g/cc)	0.021	0.043	0.007	0.014	0.010	0.020

[a]Basis 15% moisture

Table 3. Summary of Infratec Soybean Tolerance Calculation

Constituent	SEP	95% CI	0.33* SEP	95% CI	SED	95% CI SED
Moisture (%)	0.39	0.78	0.13	0.26	0.18	0.37
Protein [a] (%)	0.56	1.13	0.19	0.37	0.26	0.53
Oil [a] (%)	0.47	0.93	0.15	0.31	0.22	0.43
Fiber [a] (%)	0.09	0.19	0.03	0.06	0.04	0.09

[a]Basis 13% moisture

Table 4. Summary of Bruins/Dickey John Omega G Corn Tolerance Calculation

Constituent	SEP	95% CI	0.33* SEP	95% CI	SED	95% CI Diff.
Moisture (%)	0.56	1.12	0.19	0.37	0.26	0.52
Protein [a] (%)	0.42	0.84	0.14	0.28	0.20	0.39
Oil [a] (%)	0.41	0.82	0.14	0.27	0.19	0.38
Starch [a] (%)	0.67	1.33	0.22	0.44	0.31	0.62
Density [a] (g/cc)	0.016	0.032	0.005	0.011	0.008	0.015

[a]Dry Basis

Table 5. Summary of Bruins/Dickey John Omega G Soybean Tolerance Calculation

Constituent	SEP	95% CI	0.33* SEP	95% CI	SED	95% CI Diff.
Moisture (%)	0.39	0.78	0.13	0.26	0.18	0.36
Protein [a] (%)	0.55	1.10	0.18	0.36	0.26	0.51
Oil [a] (%)	0.43	0.86	0.14	0.28	0.20	0.40
Fiber [a] (%)	0.07	0.14	0.02	0.05	0.03	0.07

[a]Basis 13% moisture

Table 6. Summary of Perten DA7200 Corn Tolerance Calculation

Constituent	SEP	95% CI	0.33* SEP	95% CI	SED	95% CI Diff.
Moisture (%)	0.34	0.68	0.11	0.22	0.16	0.32
Protein [a] (%)	0.42	0.83	0.14	0.27	0.19	0.39
Oil [a] (%)	0.41	0.81	0.13	0.27	0.19	0.38
Starch [a] (%)	0.78	1.56	0.26	0.51	0.36	0.73
Density [a] (g/cc)	0.012	0.024	0.004	0.008	0.006	0.011

[a]Dry Basis

Table 7. Summary of Perten DA7200 Soybeans Tolerance Calculation

Constituent	SEP	95% CI	0.33* SEP	95% CI	SED	95% CI
Moisture (%)	0.43	0.86	0.14	0.28	0.20	0.40
Protein [a] (%)	0.75	1.50	0.25	0.50	0.35	0.70
Oil [a] (%)	0.44	0.88	0.15	0.29	0.21	0.41
Fiber [a] (%)	0.08	0.16	0.03	0.05	0.04	0.07

[a]Basis 13% moisture

The SEP/SECV did not show big changes when calibration were updated or validated. The accuracy and precision calculated in ISU-GQL were compared to the values calculated by NTEP (National Type Evaluation Program), published by National Conference on Weights and Measures. The NTEP validation is done on prescreened samples; no sample screening is used in the ISU-GQL samples set. The comparison is in tables 8 and 9 for corn and soybeans, respectively. Most of accuracy and precision values calculated by ISU-GQL were smaller than values from NTEP; however some constituents from Infratec and Perten instruments show larger accuracy and precision values than NTEP.

Table 8. Comparison of NTEP and ISU-GQL Standard Errors (corn)

Corn	**SEP (Accuracy)**				**Precision/ Repeatability**			
	NTEP[a]	GQL Infratec[a]	GQL Bruins[a]	GQL Perten[a]	NTEP[a]	GQL Infratec[a]	GQL Bruins[a]	GQL Perten[a]
Protein (%)	0.50	**0.54**	0.42	0.42	0.25	0.18	0.14	0.14
Oil (%)	0.50	0.47	0.41	0.41	0.20	0.15	0.14	0.13
Starch (%)	1.00	0.92	0.67	0.78	0.30	**0.31**	0.22	0.26

[a]Dry Basis

Table 9. Tolerance Comparison of NTEP and ISU-GQL Standard Errors (soybean)

Soybean	**SEP (Accuracy)**				**Precision/ Repeatability**			
	NTEP[a]	GQL Infratec[a]	GQL Bruins[a]	GQL Perten	NTEP[a]	GQL Infratec[a]	GQL Bruins[a]	GQL Perten
Protein (%)	0.55	**0.56**	0.55	**1.50**	0.25	0.19	0.18	**0.50**
Oil (%)	0.45	**0.47**	0.43	**0.88**	0.20	0.15	0.14	**0.29**

[a]Basis 13% moisture

CHAPTER 4. ONGOING QUALITY CONTROL PROGRAM FOR NIR ANALYZERS, SUPPORTING EQUIPMENT AND LABORATORY ROOM CONDITIONS

The quality control program for NIRS Analyzers consists of three activities: NIRS daily check sample, online NIRS instrument duplicates and original NIRS vs. reference chemistry. The flowchart of these activities is described in figure 3. Activities are explained below:

NIRS Daily Check Sample

The NIRS daily check sample is done to measure the precision and reproducibility of each calibrated NIRS instrument. This activity is done on constant moisture basis, 15% for corn and 13% for soybeans. The Daily NIRS check sample is done on all brands of NIRS instrument that have working calibrations. The list of NIRS instruments is shown in table 10. One sample each of corn and soybeans are used in this activity. The samples are refreshed or changed in late August. The samples are measured twice at the start of each working day. The daily check data are recorded manually in notebooks at the time of measurement and electronically in a spreadsheet every week.

Control charts are built for corn protein, oil, starch, density and for soybeans protein and oil. The first control charts used are the shewhart control charts based on mean. These control charts have control limits which are used as criteria for action. It means that when 95% confidence level is used, the control limits are mean ±2 standard deviation of the data By using this limit, there is a 5% chance that a result will exceed the control limits (Garfield et al, 2000). The control charts are created in the spreadsheet. The mean and standard deviation of measured values are calculated using functions provided by the spreadsheet software. However, the mean and standard deviation can be calculated manually using equation 1 and 2, respectively.

Table 10. List of NIRS instruments in the ISU-GQL Quality Control Program

Instrument, serial	Manufacturer	Principle	Calibration	Sample size (g)	Test cycle (min)	Wave-length (nm)	Last New Cal
Infratec 1225, 0065	Foss-Tecator	Transmittance	ANN	400	1.5	850-1048 by 2	2004
Infratec 1229, 243108	Foss-Tecator	Transmittance	ANN	400	1.5	850-1048 by 2	2004
Infratec 1229, 553075	Foss-Tecator	Transmittance	ANN	400	1.5	850-1048 by 2	2004
Infratec 1241, 12410350	Foss-Tecator	Transmittance	ANN	500	2	850-1048 by 2	2004
Omega G, 106118	Bruins GMBH	Transmittance	PLS	500	2	730-1100 by 0.5	2004
Omega AC, 301002	Bruins GMBH	Transmittance	PLS	500	2	730-1100 by 0.5	2004
Omega G, 106110	Bruins GMBH	Transmittance	PLS	500	2	730-1100 by 0.5	2004
Omega S, 201101	Bruins GMBH	Transmittance	PLS	500	2	730-1100 by 0.5	2004
Perten DA 7200, 043138	Perten Inst.	Reflectance	PLS	250	0.5	960-1650 by 5	2005
Zeltex 800, 16125	Zeltex	Transmittance	MLR	200	1.5	918-1045	2004
Zeltex 800, 16131	Zeltex	Transmittance	MLR	200	1.5	918-1046	2004
Zeltex 800, 16179	Zeltex	Transmittance	MLR	200	1.5	918-1047	2004
NIR System 6500, 3117	Foss-NIRS	Reflectance	PLS	50	2	400-2500 by 0.5	2004

ANN – Artificial Neural Network
PLS – Partial Least Squares
MLR – Multiple Linear Regressions

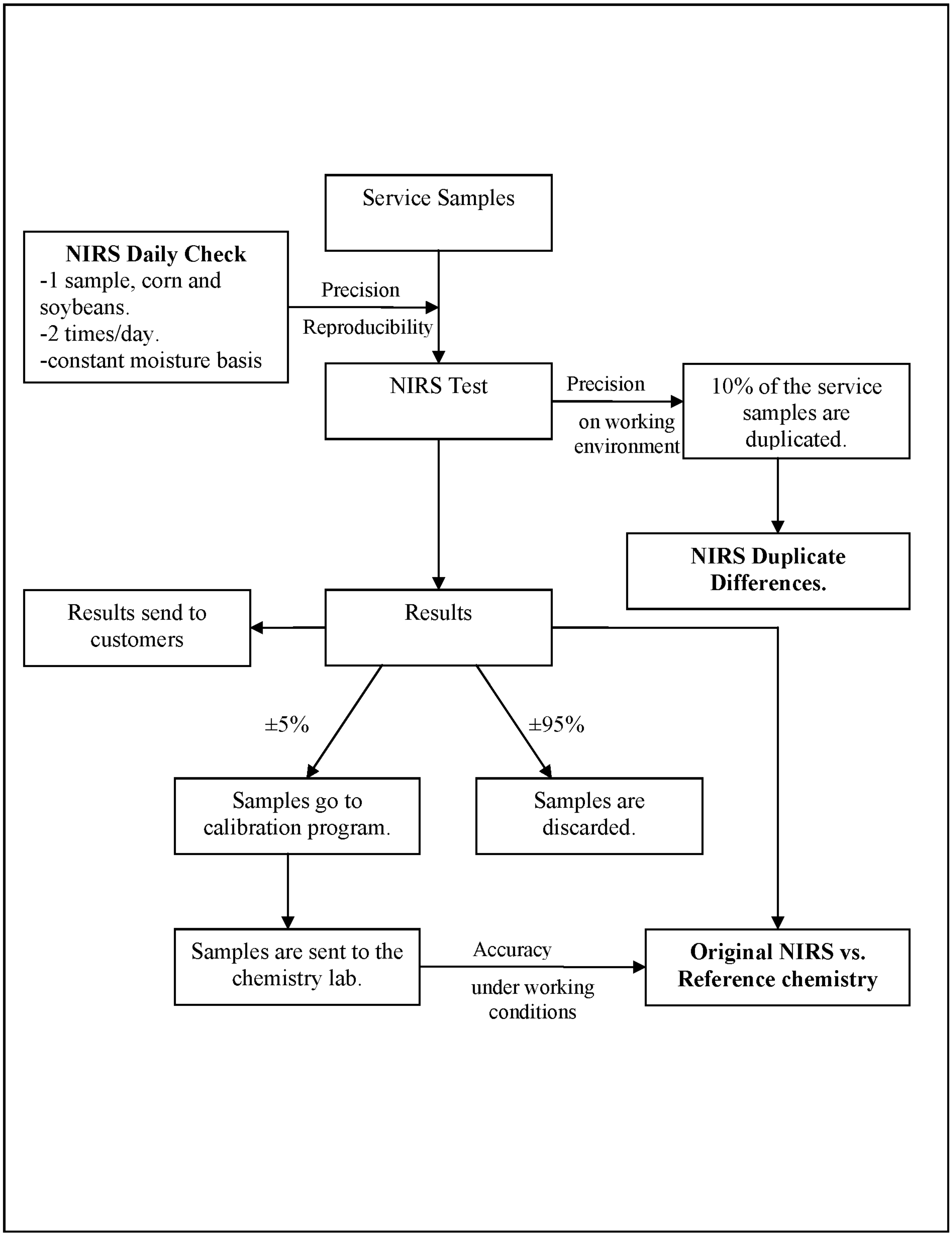


Figure 3. NIRS Analyzer Quality Control Program Flowchart

The second control chart utilizes the precision values calculated from 33% of cumulative SEP as explained in the previous chapter. This control chart uses a 10-point moving average of the data as mean or central limit of each point. A 10 point-moving average corresponds to one week of daily check data. When 95% confidence level is used, the upper and lower control limits are a 10-point moving average ±2 *(0.33 SEP). The SEP(s) are calculated based on cumulative average of SECV and SEP as explained in Chapter 3. By using this control chart, every data point has its own control limits.

A better method of control chart will be chosen. Every instrument has its own control chart. The control charts are printed and put in the respective NIRS instrument notebook biweekly. The report for this activity is updated quarterly with summary of average, standard deviation, number of readings, maximum and minimum values, and percentage of out of control data of each instrument. Online control may be applied, but it has some difficulties. Employees sometimes confuse the quality control files with other files and enter the data in the wrong files. Since the data have been recorded in the notebooks, documentation is maintained. When the notebooks are used to record analytical results for samples, the laboratory will need to establish a standardized notebook control procedures (Garfield et al, 2000). ISU-GQL has standardized the notebooks used for recording NIRS daily check data according to ISO17025 requirements.

Online NIRS Instrument Duplicates

The NIRS instrument duplicates are done to check the precision of the NIRS instruments under working conditions. The instrument duplicates are done when ISU-GQL does service for customers. Ten percent of NIRS instrument readings are duplicated. The differences are calculated from the samples and their duplicates. A control chart is built to evaluate whether the instruments are able to do the measurement precisely over many different samples, more than the single daily check. Two methods of control chart will be compared.

The first control chart is a shewhart control chart based on mean of the differences with 95% confidence level. This corresponds to mean of the differences ±2 standard deviation of differences. The second control chart is built based on the assumption that the differences between pairs follow a Normal Distribution with mean zero and standard error of difference $\sqrt{2}$ *0.33 SEP as explained in previous chapter. The 95% confidence level is also used in this chart: the tolerance then becomes $\pm 2 * \sqrt{2} * 0.33$ SEP. These limits are fixed and do not depend on the data, like the shewhart control chart. The tolerance values for the differences between pair of each instrument have been calculated in the previous chapter.

A better method of control chart will be chosen. The duplicate difference control charts are printed and put in the instrument notebook every two weeks. A Report for this activity is updated quarterly with summary of average and standard deviation of differences, number of samples and percentage of out of control data of each instrument. The differences from previous year results are also summarized.

Online warnings are possible with instrument duplicates. The additional columns that have been formatted are added to the service spreadsheet. When the difference between first and second run is not between the upper and control limits, the additional column shows "NOT OK". The quality control manager will handle this problem.

Original NIRS vs. references chemistry

Comparison between the original NIRS values and reference chemistry is done to measure the working accuracy of NIRS instrument. The original NIRS results of samples chosen for calibration (x-axis) are plotted against the reference chemistry values (y-axis), with the y=x line is the center limit. . A reference chemistry calibration check is done annually. The upper and lower control limit using 95% confidence level are equal to $y = x \pm 2$ SEP, since SEP measures the accuracy of NIRS instrument with the respect to reference data. SEP(s) were calculated based on procedures in Chapter 3. The SEP values of every instrument brand are also available in the previous chapter. The number of samples, R^2 and standard error of every instrument used for the service are summarized for the yearly report. The charts are printed and put in the instrument notebook every year.

Supporting Equipment

Seven balances are checked yearly for a full range and monthly at a single point. The measurement in each balance is done in three times. The percentages of differences from the true values are calculated and the results are plotted in a chart. When the chart of differences between measured and true values show the trends of increasing error, the balance may have problem.

Two types of thermometers (infrared and mercury) are checked monthly. The mercury thermometer (NIST calibrated) is used as a reference to check an infrared thermometer from Fisher Scientific (Fisher Scientific, www. fishersci.com). Room, refrigerator and boiling water temperature are measured three times in the monthly thermometer test. The differences of IR readings from mercury thermometer are calculated and plotted on the control chart with upper and lower control limits are $\pm 2^0$C (these values are accuracy stated by the factory). When the control chart shows more than 5% out of control data, the infrared thermometer may need to change battery and /or be recalibrated.

The seed counter Syntron Magnetic Parts Feeder EB-00 (Seedburo Equipment Company, www.seedburo.com) is checked yearly for full scale counts (100, 200, 300, 400 and 500 seeds) and weekly for a single point (300 seeds). Corn and soybean samples are used in this activity. The percentages of differences from the true values are calculated. The 95% and 99% confidence level of shewhart control charts which correspond to warning and action limits are constructed for the percentage of differences from the true values for each corn and soybeans sample. When the control chart shows more than 5% and/or 1% out of control data at the warning and action limits, respectively, the seed counter may have a problem with either the operator or the equipment. However, problem may come from untrained operator, if this happen then operator training is needed.

Two dividers (Rotary and Boerner) are checked monthly. Three measurements are done in each test. The Rotary divider divides the sample into 10%, 20%, 30% and 40% portions. The Boerner divider divides samples into equal portions. A 1000 – gram corn sample is used for the divider checks. The differences from the true portions are calculated. The 95% and 99% confidence level of shewhart control charts which correspond to warning and action limits is constructed for the differences from the true portion for each divider. When the control chart shows more than 5% and/or 1% out of control data at the warning and action limits, respectively, dividers may have problem or operator training is needed.

The grain test weight is done with the kettle (quart and pint) and two GAC instruments (GAC 2000 and GAC 2100). GAC instruments also measure the sample moisture. The test weight results are corrected for moisture using the formula below:

Test weight corrected = Test weight original + (0.25* (current moisture – 15%)

(eq.6) (Iowa State University Extension, 1998).

The purpose of correcting test weight is to reduce differences from changes in moisture. The grain test weight and divider check use the same corn sample. This sample is changed at least every year. There are five charts built for this activity:

- Date vs. test weight using kettle (quart and pint).
- Date vs. test weight using GAC 2000 and GAC 2100.
- 95% control chart of differences between average of test weight (quart and pint) and average of test weight from GAC 2000 and GAC 2100.
- Test weight of kettle (quart and pint) vs. GAC 2000/2100.

A water test weight check is done annually on quart and pint. The purpose of performing water test weight is to know whether quart and pint have the right volume according to the standard volume: 1098.10 gram for quart and 549.05 gram for pint. USDA Equipment Handbook suggests the tolerance for the water test weight is ±1 gram. (USDA Equipment Handbook, 1996)

All data from supporting equipment check are recorded manually in the lab QC book and electronically in the spreadsheet. Charts are printed and put in the lab QC book monthly. The supporting equipment quality control results are reported quarterly.

Laboratory room conditions

The laboratory climate conditions (temperature, relative humidity and barometric pressure) are monitored by climate sensor. This climate sensor is connected to the computer. The C3DAS Data Acquisition software is able to capture climate data every hour. The climate data are downloaded monthly to a spreadsheet. Monthly charts that plot hourly time against temperature, humidity and pressure are created. Any of the instrument control operations can be referenced against the room air conditions (eg. NIRS daily check sample).

The data and control charts of every quality control activity are documented for future use. Procedures for operating QC, handling and documenting data, developing and updating control charts were written for each QC activity. These procedures can be seen in Appendix I. The quality control programs for NIRS instruments, supporting equipment and laboratory room conditions are summarized in the tables 11, 12, and 13.

Table 11. NIRS Quality Control Program

Item	Check	Quality Control	Current Tolerance	Documentation	Comments
NIRS Daily Check **Infratec** Infratec 1225, 0065 Infratec 1229, 243108 Infratec 1229, 553075 Infratec 1241, 12410350	Precision	95% Confidence Level of 10-point moving average chart	Allow 5% out of control data **Corn**: Protein (mean ± 0.30); Oil (mean ± 0.26); Starch (mean ± 0.52); Density (mean ± 0.014) **SB**: Protein (mean ± 0.37); Oil (mean ± 0.31); Fiber (mean ± 0.06) mean=10-point moving average M,P,O,S,F = %, D = g/cm3	Lab notebooks (every day) Spreadsheet (every week)	One sample per grain, run daily (corn and soybean) Biweekly printed control chart Quarterly updated report (summary of ave, std.dev, max, min, count and percentage of out of control data) Yearly Updated Tolerances
Bruins Omega G, 106118 Omega AC, 301002 Omega G, 6110 Omega S, 201101	Precision	95% Confidence Level of 10-point moving average chart	Allow 5% out of control data **Corn**: Protein (mean ± 0.28); Oil (mean ± 0.27); Starch (mean ± 0.44); Density (mean ± 0.011) **SB**: Protein (mean ± 0.36); Oil (mean ± 0.28); Fiber (mean ± 0.05) mean =10-point moving average M,P,O,S,F = %, D = g/cm3	Lab notebooks (every day) Spreadsheet (every week)	One sample per grain, run daily (corn and soybean) Biweekly printed control chart Quarterly updated report (summary of ave, std.dev, max, min, count and percentage of out of control data) Yearly Updated Tolerances
Perten Perten DA 7200 43138	Precision	95% Confidence Level of 10-point moving average chart	Allow 5% out of control data **Corn**: Protein (mean ± 0.27); Oil (mean ± 0.27); Starch (mean ± 0.51); Density (mean ± 0.008) **SB**: Protein (mean ± 0.50); Oil (mean ± 0.29); Fiber (mean ± 0.05) mean =10-point moving average M,P,O,S,F = %, D = g/cm3	Lab notebooks (every day) Spreadsheet (every week)	One sample per grain, run daily (corn and soybean) Biweekly printed control chart Quarterly updated report (summary of ave, std.dev, max, min, count and percentage of out of control data) Yearly Updated Tolerances

Table 11. Continued

Item	Check	Quality Control	Current Tolerance	Documentation	Comments
Zeltex Zeltex 800, 16125 Zeltex 800, 16131 Zeltex 800, 16179	Precision	95% Confidence level of Shewhart control chart	Allow 5% out of control data	Lab notebooks (every day) Spreadsheet (every week)	One sample per grain, run daily (corn and soybean) Biweekly printed control chart Quarterly updated report (summary of ave, std.dev, max, min, count and percentage of out of control data)
NIR System 6500 3117	Precision	95% Confidence level of Shewhart control chart	Allow 5% out of control data	Lab notebooks (every day) Spreadsheet (every week)	One sample per grain, run daily (corn and soybean) Biweekly printed control chart Quarterly updated report (summary of ave, std.dev, max, min, count and percentage of out of control data)
NIRS Duplicates Infratec 1225, 0065 Infratec 1229, 243108 Infratec 1229, 553075 Infratec 1241, 12410350	Precision	95% Confidence Level control chart of duplicates differences (10% NIR readings) Summary of average and std. dev of differences every instrument (every year) Summary of differences from previous year (ave, std.dev)	Allow 5% out of control data **Corn**: Moist (mean ± 0.51); Protein (mean ± 0.43); Oil (mean ± 0.37); Starch (mean ± 0.73); Density (mean ± 0.020) **SB**: Moist (mean ± 0.37); Protein (mean ± 0.53); Oil (mean ± 0.43); Fiber (mean ± 0.09) mean difference between duplicates = 0 M,P,O,S,F = %, D = g/cm3	Spreadsheet	Every 10th sample, service test Biweekly printed control chart Quarterly updated report (summary of ave and std.dev of differences, number of samples and percentage of out of control data) Yearly updated tolerances
NIRS comparison to references Infratec 1225, 0065 Infratec 1229, 243108 Infratec 1229, 553075 Infratec 1241, 12410350	Accuracy	95% Confidence level Chart of Original NIR predictions vs. reference chemistry values Summary of R^2 and standard error of every instrument	Allow 5% out of control data **Corn**: Protein (NIR ± 0.92); Oil (NIR ± 0.80); Starch (NIR ± 1.56); Density (NIR ± 0.043) **SB**: Protein (NIR ± 1.13); Oil (NIR ± 0.93) NIR = original prediction values M,P,O,S,F = %, D = g/cm3	Spreadsheet	During calibration About 150 calibration samples Yearly printed chart Yearly updated report (summary of number of samples, R^2 and standard error of each instrument) Yearly updated tolerances

Table 12. Supporting Equipment Quality Control Program

Item	Check	Quality Control	Tolerance	Documentation	Comments
Equipment Check					
Balances (7) AND HR-60 Denver A-250 Mettler AJ 100 Mettler PB 153-S Mettler PM- 4000 Mettler SB-16000 Seed Burro 8800	Accuracy	Chart of Date vs. Error Percentages.	Follow factory specification	Lab notebooks Spreadsheet	Yearly full scale check Monthly single point check Monthly printed chart Quarterly updated report
Thermometers (2) Infra Red Mercury	Accuracy	Control chart of the differences between Infrared and Mercury thermometer with UCL/LCL= $\pm 2^0$C	Allow 5% out of control data	Lab notebooks Spreadsheet	Monthly single point check Monthly printed control charts Quarterly updated report
Divider (2) Rotary Boerner	Accuracy	95% and 99% CL shewhart control chart of error percentages	Allow 5% and 1% out of control data respectively.	Lab notebooks Spreadsheet	Monthly check Monthly printed control charts Quarterly updated report
Seed counter (1)	Accuracy	95% and 99% CL shewhart control chart of error percentages Corn and soybeans	Allow 5% and 1% out of control data respectively.	Lab notebooks Spreadsheet	Yearly full scale check Single point weekly check (300) Monthly printed control charts Quarterly updated report
Periodic Check **Grain Test Weight (kettle and GAC)**	Accuracy	Chart Date vs. TW Quart/Pint Chart Date vs. TW GAC 2000/2100 Chart Date vs. Moist. GAC 2000/2100 95% CL of shewhart control chart (Differences between Quart/Pint and GAC 2000/2100 TW) Chart TW Quart/Pint vs. GAC 2000/2100	Allow 5% out of control data	Lab notebooks Spreadsheet	Weekly check Quarterly printed control charts Quarterly updated report
Water Test Weight	Accuracy	Control chart of the differences between measured and actual weight. UCL/LCL= ± 1 gram	Allow 5% out of control data	Lab notebooks Spreadsheet	Yearly check Yearly printed control charts Yearly updated report

Table 13. Laboratory Room Conditions Quality Control

Item	Check	Quality Control	Tolerance	Documentation	Comments
Climate					
Temperature	fluctuation	Chart of time vs. temperature		Spreadsheet	Hourly data captured
Humidity		Chart of time vs. humidity			Monthly data download
Pressure		Chart of time vs. pressure			

CHAPTER 5. IMPLEMENTATION OF THE ONGOING QUALITY CONTROL PROGRAM FOR NIRS ANALYZERS, SUPPORTING EQUIPMENT AND LABORATORY ROOM CONDITIONS

This chapter describes the implementation of an ongoing quality control program for NIRS analyzers, supporting equipment and laboratory room conditions. Data from initial application are given.

NIRS Daily Check Sample

The examples of NIRS daily check implementation were taken from 9/12/2005 to 3/8/2006 for corn (sample 20040442) and 3/1/2005 to 3/8/2006 for soybeans (sample 20010461). However, there were some irregularities in doing this activity. For example, the employee who did daily check sample was not able to come because of sickness or school holidays or instruments were used for other activities. These irregularities should be recorded in the instrument notebook. This is one of the challenges that university laboratories face since they employ students that are only able to work part time.

Tables 14 and 15 summarize the results of soybean protein and oil NIRS daily checks. The first method which uses mean ± 2 standard deviation of the data shows that on average, the percentages out of control data of NIRS instruments are about 5% which agrees to the definition of 95% confidence level. The second method which tracks every single data with 10-moving average ± 2 (0.33 SEP) shows that out of control data each instrument varied from 5% to 30%. The 95% confidence level of 0.33 SEP for each instrument used values in tables 3 and 5 calculated in Chapter 3. The SEP data for NIR Systems 6500 and Zeltex 800 were not available; therefore the second method could not be performed. Currently, Perten DA 7200 only does daily check using corn sample.

Table 14. Soybean Protein NIRS Instrument Daily Check

Instrument	Ave (%)	Std. Dev (%pts)	Min (%)	Max (%)	Range (% pts)	Count	Out of Control Data	
							Method 1	Method 2
Infratec 1225 0065	38.28	0.74	37.00	42.20	5.20	334	5.99%	30.46%
Infratec 1229 243108	37.91	0.27	37.20	38.60	1.40	330	5.15%	10.59%
Infratec 1229 553075	38.62	0.82	37.40	40.30	2.90	330	0.91%	6.54%
Infratec 1229 553792	38.08	0.31	36.20	39.00	2.80	206	3.88%	10.15%
Infratec 1241 12410350	37.98	0.20	37.10	38.60	1.50	307	5.21%	3.69%
NIR System 6500 3117	38.80	1.87	35.29	57.15	21.86	295	0.68%	n/a
Omega AC 301002	37.89	0.30	36.00	38.60	2.60	327	3.98%	12.26%
Omega G 106110	38.17	0.30	37.30	38.70	1.40	291	3.78%	8.87%
Omega G 106118	38.04	0.28	37.00	39.30	2.30	315	3.49%	10.46%
Omega S 201101	37.85	0.39	36.60	40.60	4.00	279	5.02%	23.70%
Perten DA 7200 043138	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
Zeltex 800 16125	38.91	1.30	33.88	41.88	8.00	299	7.36%	n/a
Zeltex 800 16131	44.05	1.76	40.09	48.58	8.49	293	1.37%	n/a
Zeltex 800 16179	42.68	1.51	39.35	46.33	6.98	292	2.74%	n/a

Table 15. Soybean Oil NIRS Instrument Daily Check

Instrument	Ave (%)	Std. Dev (%pts)	Min (%)	Max (%)	Range (% pts)	Count	Out of Control Data	
							Method 1	Method 2
Infratec 1225 0065	18.83	0.30	17.40	19.60	2.20	334	3.89%	14.15%
Infratec 1229 243108	18.86	0.20	18.00	19.80	1.80	330	3.64%	6.54%
Infratec 1229 553075	19.28	0.46	18.00	20.20	2.20	330	0.61%	5.30%
Infratec 1229 553792	19.00	0.16	18.60	19.90	1.30	206	1.94%	2.54%
Infratec 1241 12410350	18.91	0.20	18.10	19.90	1.80	307	3.23%	3.36%
NIR System 6500 3117	19.46	0.45	18.17	22.65	4.48	294	2.34%	n/a
Omega AC 301002	19.01	0.22	17.30	19.60	2.30	327	3.06%	10.06%
Omega G 106110	19.42	0.27	18.40	19.90	1.50	291	6.87%	15.60%
Omega G 106118	19.24	0.18	18.40	19.90	1.50	315	4.76%	7.84%
Omega S 201101	18.98	0.33	17.70	20.90	3.20	279	5.38%	28.52%
Perten DA 7200 043138	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
Zeltex 800 16125	20.45	0.56	18.27	21.70	3.43	299	4.01%	n/a
Zeltex 800 16131	16.29	0.70	14.67	18.39	3.72	293	2.73%	n/a
Zeltex 800 16179	17.63	19.61	16.11	0.59	3.50	292	4.79%	n/a

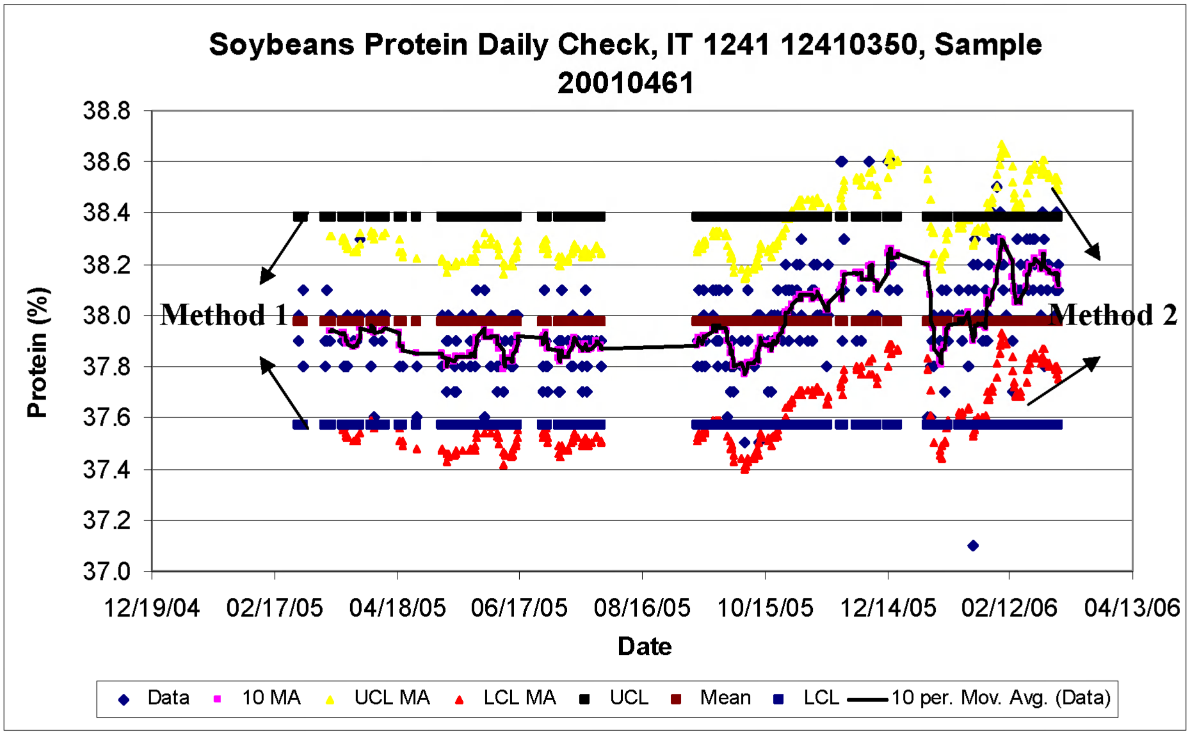


Figure 4. Soybeans Protein Daily Check, Sample 20010461, IT 1241 12410350 Control Chart

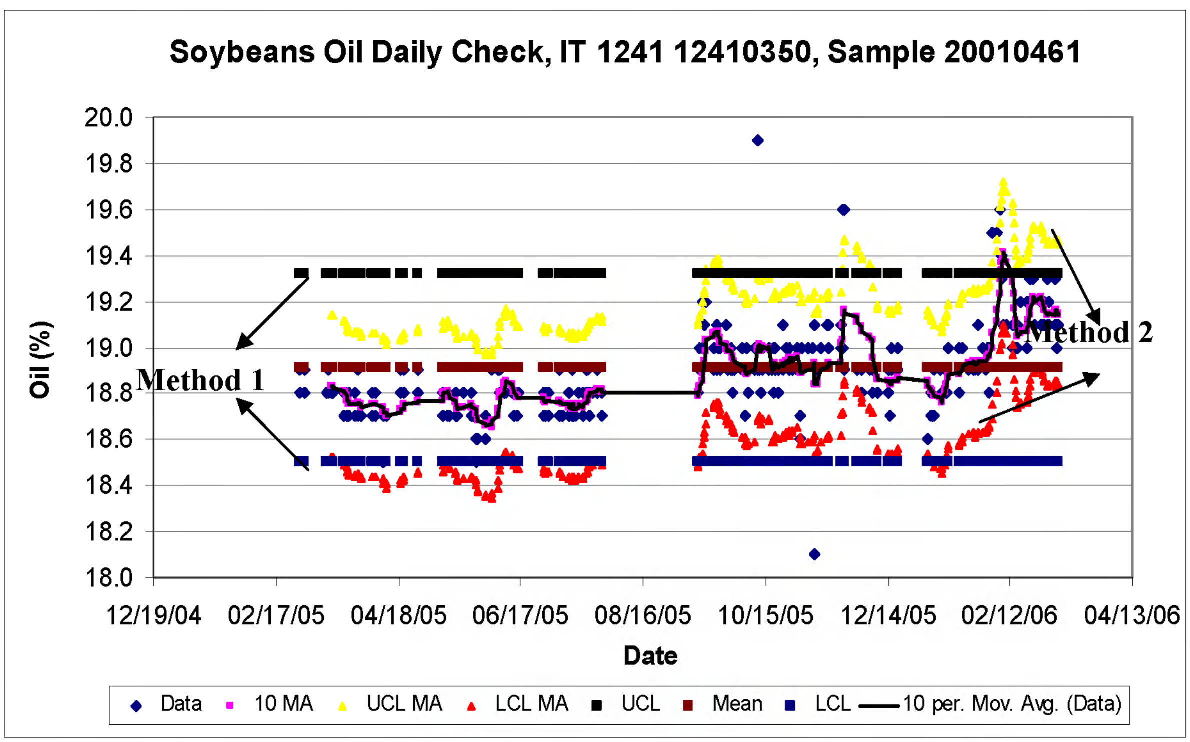


Figure 5. Soybeans Oil Daily Check, Sample 20010461, IT 1241 12410350 Control Chart

Figures 4 and 5 are the examples of daily check control chart for soybeans protein and oil from Infratec 1241 12410350 instrument. The control charts for other instruments can be seen in the Appendix B. From the chart, control limits from first method cannot track individual data and will get bigger once the standard deviation gets bigger. The bad data protect themselves. The second method gives tighter control than the first method. It can track every single data and follow trends of the data. This method can give control even with the cyclic pattern that appears in the NIR daily check. The reason for the cyclic patterns is not known.

Tables 16, 17, 18 and 19 recap the NIRS daily check results for corn protein, oil, starch and density from all instruments. The first method shows that the percentages of out of control data for all instruments are about 5%. The second method shows that out of control data of each instrument is generally larger than 5%, except for corn density. The high percentage of out of control data can be caused by many factors such as temperature/humidity fluctuation, untrained operators, and sample physical properties changes. The exact causes may need to be determined.

Figures 6, 7, 8 and 9 are the examples of control chart for corn protein, oil, starch and density from Infratec 1241 12410350 instrument. The corn control charts for other instruments can be seen in the Appendix B.

Table 16. Corn Protein NIRS Instrument Daily Check

Instrument	Ave (%)	Std. Dev (%pts)	Min (%)	Max (%)	Range (% pts)	Count	Out of Control Data	
							Method 1	Method 2
Infratec 1225 0065	6.38	0.51	5.50	8.20	2.70	198	6.06%	30.69%
Infratec 1229 243108	6.38	0.45	5.60	8.30	2.70	194	5.67%	31.35%
Infratec 1229 553075	6.36	0.51	5.20	8.40	3.20	202	6.44%	31.09%
Infratec 1229 553792	6.44	0.53	5.50	8.60	3.10	170	8.24%	30.63%
Infratec 1241 12410350	7.69	0.48	7.00	9.80	2.80	193	5.70%	26.09%
NIR System 6500 3117	5.41	0.57	3.96	8.87	4.91	164	6.10%	n/a
Omega AC 301002	7.09	0.77	5.50	9.40	3.90	199	6.03%	62.63%
Omega G 106110	7.57	0.68	5.30	9.70	4.40	188	7.98%	39.66%
Omega G 106118	7.46	0.64	6.00	9.50	3.50	203	6.40%	32.99%
Omega S 201101	7.23	0.98	5.00	10.90	5.90	200	5.50%	58.64%
Perten DA 7200 043138	7.48	0.57	6.00	9.40	3.40	163	4.29%	29.22%
Zeltex 800 16125	9.06	0.53	7.59	10.76	3.17	160	4.38%	n/a
Zeltex 800 16131	5.74	0.78	3.90	8.33	4.43	157	5.73%	n/a
Zeltex 800 16179	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a

Table 17. Corn Oil NIRS Instrument Daily Check

Instrument	Ave (%)	Std. Dev (%)	Min (%)	Max (%)	Range (%)	Count	Out of Control Data	
							Method 1	Method 2
Infratec 1225 0065	4.16	0.32	3.50	5.10	1.60	198	4.55%	17.99%
Infratec 1229 243108	4.14	0.26	3.70	5.10	1.40	194	3.09%	17.84%
Infratec 1229 553075	4.29	0.30	3.60	5.60	2.00	202	4.46%	17.62%
Infratec 1229 553792	4.12	0.27	3.70	5.10	1.40	170	4.71%	16.77%
Infratec 1241 12410350	5.03	0.32	4.30	6.00	1.70	193	3.11%	22.83%
NIR System 6500 3117	4.05	0.38	2.75	5.20	2.45	164	5.49%	n/a
Omega AC 301002	4.39	0.57	3.40	7.20	3.80	199	5.53%	41.58%
Omega G 106110	4.87	0.45	4.10	6.50	2.40	188	6.91%	29.61%
Omega G 106118	4.82	0.48	4.00	6.60	2.60	203	5.91%	30.93%
Omega S 201101	4.65	0.66	3.20	7.10	3.90	200	6.00%	47.12%
Perten DA 7200 043138	4.68	0.29	4.10	5.90	1.80	163	3.68%	20.13%
Zeltex 800 16125	5.93	0.67	3.83	7.51	3.68	160	4.38%	n/a
Zeltex 800 16131	5.18	0.73	3.88	6.97	3.09	157	4.46%	n/a
Zeltex 800 16179	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a

Table 18. Corn Starch NIRS Instrument Daily Check

Instrument	Ave (%)	Std. Dev (%pts)	Min (%)	Max (%)	Range (% pts)	Count	Out of Control Data Method 1	Method 2
Infratec 1225 0065	61.05	1.05	57.20	63.50	6.30	198	7.07%	17.99%
Infratec 1229 243108	60.88	0.90	57.00	62.60	5.60	194	4.64%	17.84%
Infratec 1229 553075	61.01	0.95	57.40	63.10	5.70	202	6.93%	17.62%
Infratec 1229 553792	60.89	0.90	57.70	62.50	4.80	170	6.47%	16.77%
Infratec 1241 12410350	71.69	0.99	67.90	74.40	6.50	193	7.77%	22.83%
NIR System 6500 3117	61.64	0.71	58.89	63.89	5.00	163	5.52%	n/a
Omega AC 301002	73.19	1.40	65.60	78.40	12.80	199	4.52%	41.58%
Omega G 106110	71.84	1.05	68.50	75.50	7.00	188	7.45%	29.61%
Omega G 106118	72.05	1.06	68.10	73.90	5.80	203	5.42%	30.93%
Omega S 201101	72.39	1.49	66.70	75.60	8.90	200	7.00%	47.12%
Perten DA 7200 043138	72.29	0.69	69.50	73.50	4.00	163	3.07%	20.13%
Zeltex 800 16125	58.10	0.70	56.24	60.11	3.87	160	5.00%	n/a
Zeltex 800 16131	64.13	1.26	61.28	66.77	5.49	157	3.18%	n/a
Zeltex 800 16179	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a

Table 19. Corn Density NIRS Instrument Daily Check

Instrument	Ave (g/cm^3)	Std. Dev (g/cm^3 pts)	Min (g/cm^3)	Max (g/cm^3)	Range (g/cm^3 pts)	Count	Out of Control Data Method 1	Method 2
Infratec 1225 0065	1.223	0.014	1.114	1.282	0.168	198	1.52%	3.70%
Infratec 1229 243108	1.224	0.010	1.198	1.250	0.052	194	5.67%	6.49%
Infratec 1229 553075	1.219	0.007	1.198	1.238	0.040	202	4.95%	0.52%
Infratec 1229 553792	1.229	0.007	1.209	1.250	0.041	170	1.76%	1.86%
Infratec 1241 12410350	1.226	0.010	1.202	1.256	0.054	193	4.66%	0.54%
NIR System 6500 3117	1.233	0.006	1.221	1.251	0.030	164	2.44%	n/a
Omega AC 301002	1.244	0.014	1.216	1.282	0.066	194	3.09%	10.81%
Omega G 106110	1.233	0.012	1.208	1.258	0.050	186	2.14%	10.17%
Omega G 106118	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
Omega S 201101	1.229	0.016	1.165	1.271	0.106	200	5.50%	29.84%
Perten DA 7200 043138	1.241	0.005	1.229	1.253	0.024	163	2.45%	0.65%

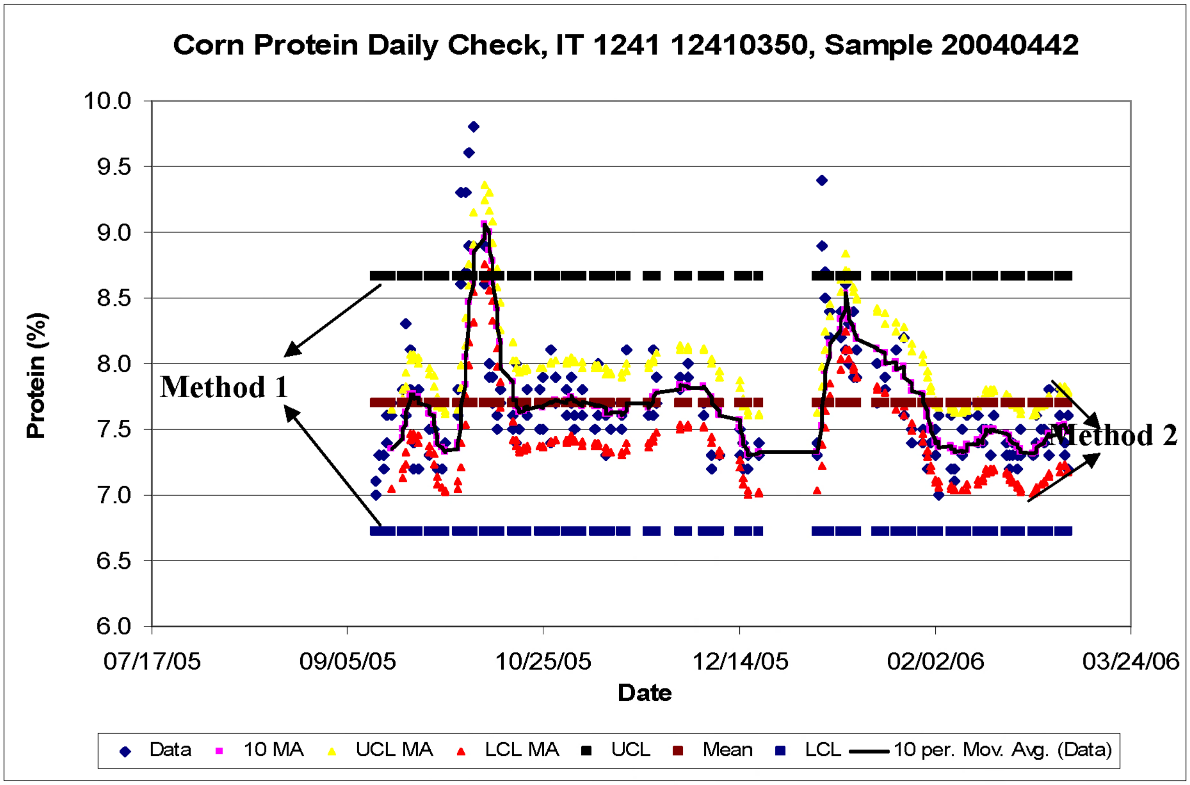


Figure 6. Corn Protein Daily Check, Sample 20040442, IT 1241 12410350 Control Chart

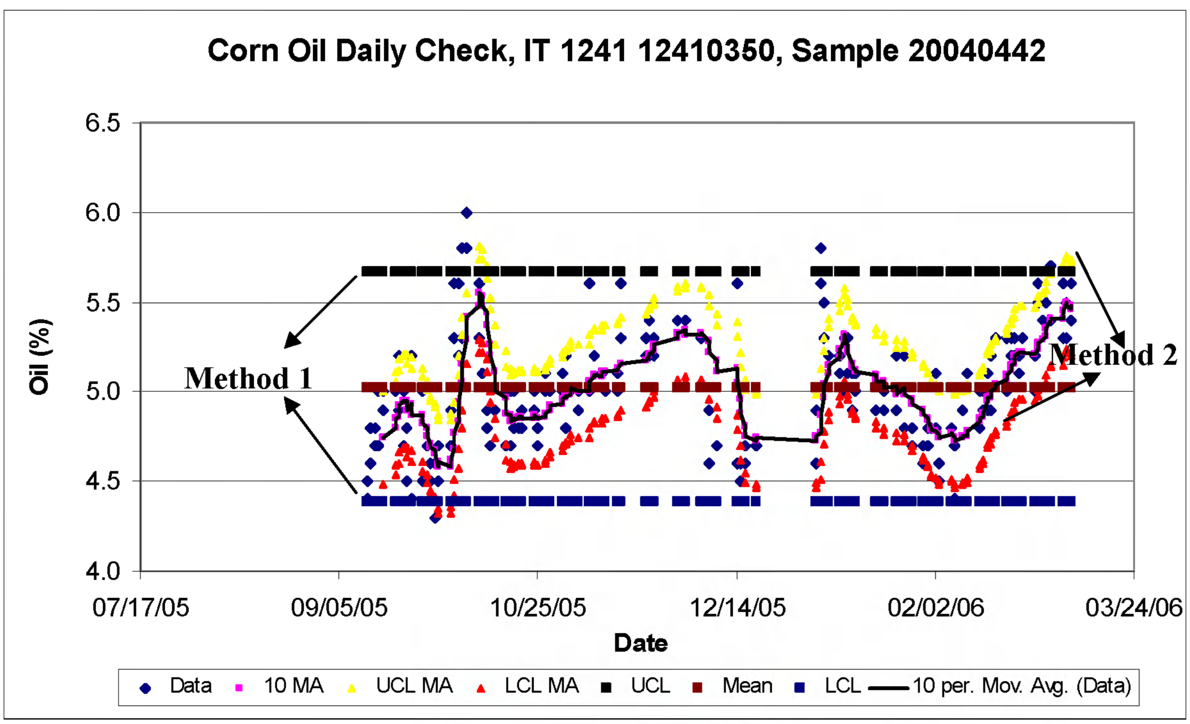


Figure 7. Corn Oil Daily Check, Sample 20040442, IT 1241 12410350 Control Chart

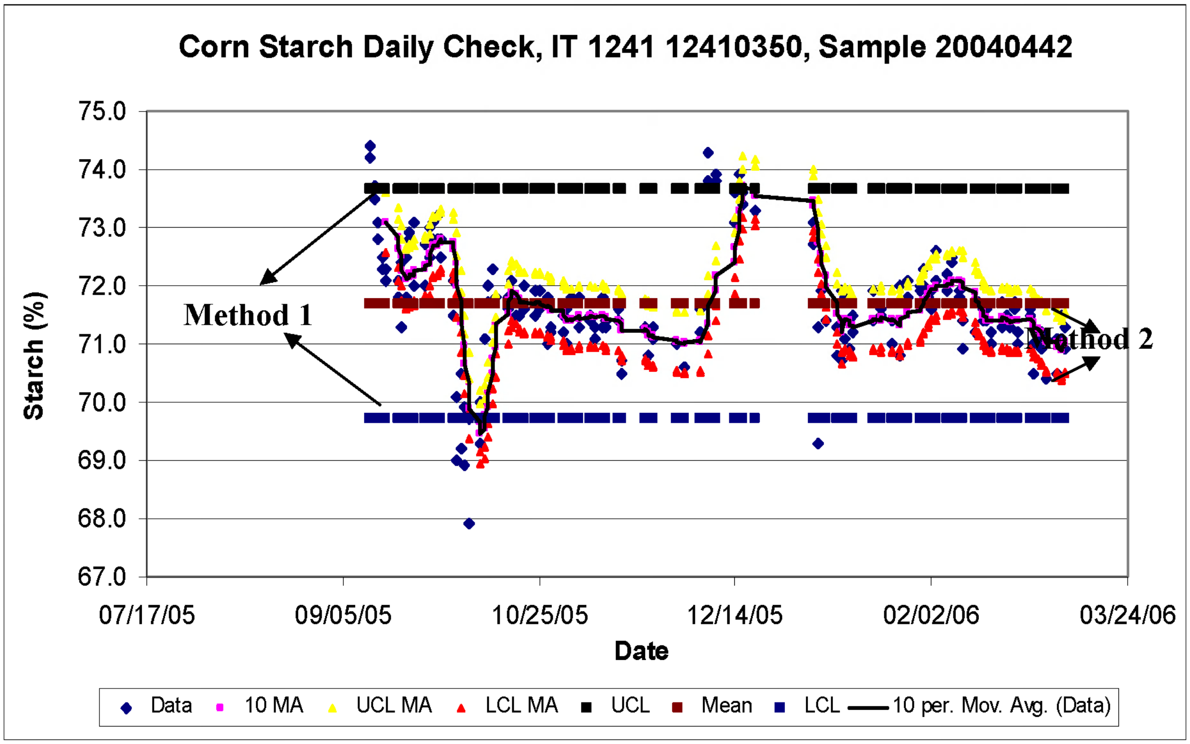


Figure 8. Corn Starch Daily Check, Sample 20040442, IT 1241 12410350 Control Chart

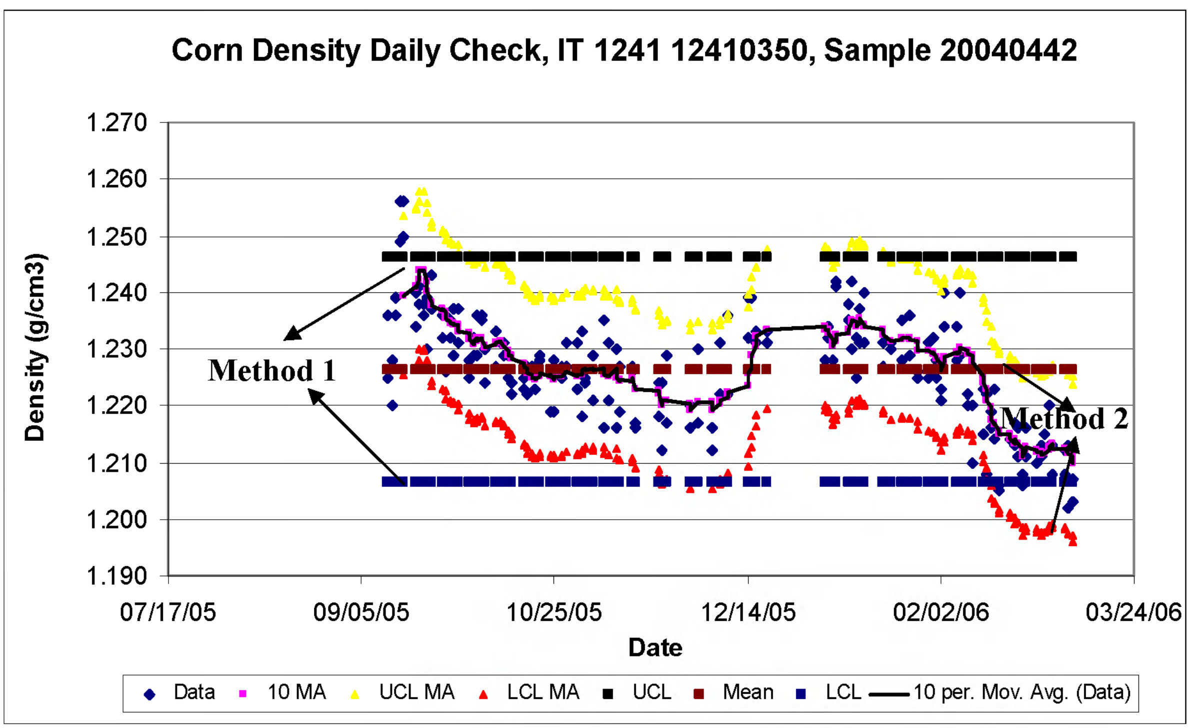


Figure 9. Corn Density Daily Check, Sample 20040442, IT 1241 12410350 Control Chart

NIRS Instrument Duplicates

Tables 20 and 21 contain summary of NIRS instrument duplicate differences from the 2005 service activity for corn and soybeans, respectively. Method 1 uses average of differences ± 2 standard deviations based on the data. On average, the out of control data are about 5% which agrees with the definition of standard deviation in the normal distribution. Method 2 uses ± $\sqrt{2}$*0.33 SEP as calculated in tables 2 and 3 in chapter 3. The out of control data vary, since the tolerance is fixed and not taken from their own data. Figures 10 and 11 are the control chart examples for NIRS duplicate differences using method 1 and method 2 that are applied to soybeans protein and corn oil, respectively. A Summary of duplicate differences from 2004 and all duplicate differences control charts for 2005 can be found in Appendix C.

Table 20. NIRS Instrument Duplicate Differences, Soybeans 2005

Factor	NIR Unit	Average of Differences	Standard Deviation of Differences	n	Out of control	
					Method 1	Method 2
Moisture (%)	IT553075	0.01	0.22	154	7.14%	9.74%
	IT243108	0.03	0.21	407	5.16%	7.62%
	IT65	-0.09	0.54	117	2.56%	10.26%
	IT 553792	0.04	0.20	359	2.79%	6.13%
Protein (%)	IT553075	0.03	0.33	154	5.19%	6.49%
	IT243108	0.00	0.27	407	4.18%	4.18%
	IT65	0.00	0.35	117	5.98%	7.69%
	IT 553792	-0.01	0.26	359	3.62%	3.62%
Oil (%)	IT553075	-0.02	0.26	154	1.95%	2.60%
	IT243108	0.00	0.21	407	4.91%	4.91%
	IT65	-0.03	0.28	117	7.69%	10.26%
	IT 553792	-0.01	0.18	359	4.18%	1.67%
Average					**4.61%**	**6.26%**

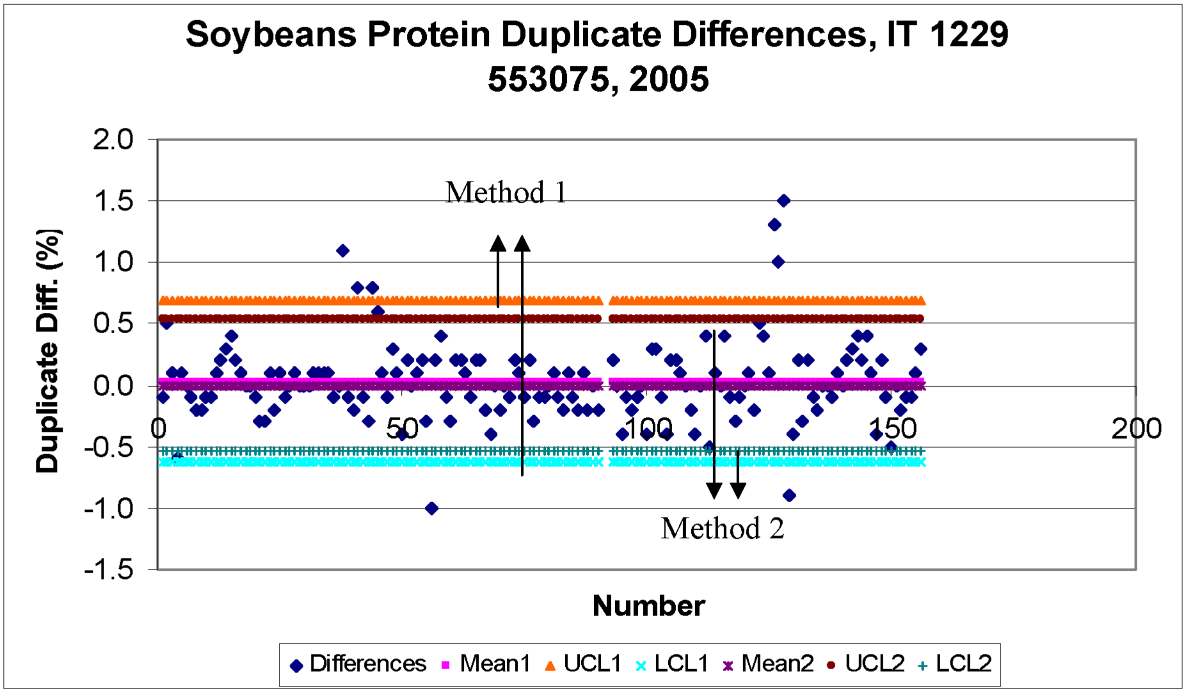


Figure 10. Soybeans Protein Duplicate Differences 2005, IT 1229 553705 Control Chart, Method 1 and 2

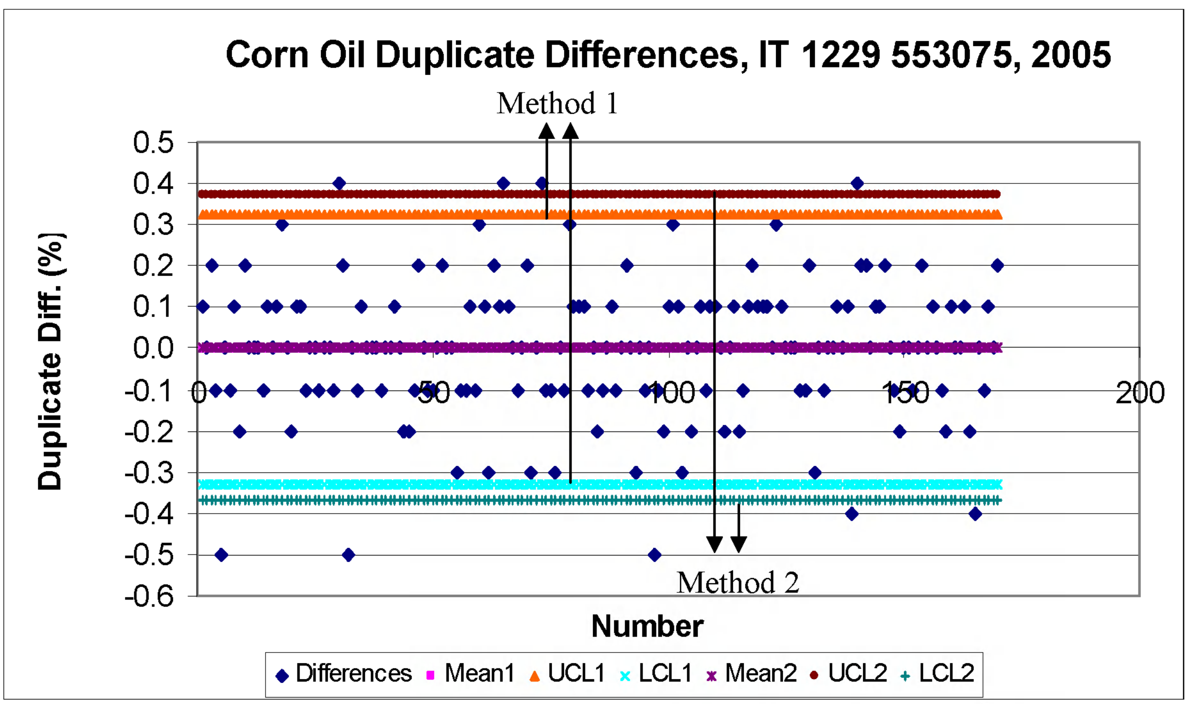


Figure 11. Corn Oil Duplicate Differences 2005, IT 1229 553075 Control Chart, Method 1 and 2

Table 21. NIRS Instrument Duplicate Differences, Corn 2005

Constituent	NIR Unit	Average of Differences	Standard Deviation of Differences	n	Out of control	
					Method 1	Method 2
Moisture (%)	IT553075	0.02	0.20	170	5.88%	2.94%
	IT243108	-0.07	0.32	29	10.34%	10.34%
	IT65	0.03	0.16	36	5.56%	0.00%
	IT 553792	0.01	0.07	18	0.00%	0.00%
Protein (%)	IT553075	-0.01	0.14	170	6.47%	0.00%
	IT243108	0.06	0.20	29	3.45%	3.45%
	IT65	0.00	0.12	36	2.78%	0.00%
	IT 553792	-0.02	0.13	18	0.00%	0.00%
Oil (%)	IT553075	0.00	0.16	170	5.29%	5.29%
	IT243108	0.03	0.14	29	3.45%	3.45%
	IT65	0.03	0.13	36	5.56%	2.78%
	IT 553792	0.02	0.17	18	5.56%	5.56%
Starch (%)	IT553075	-0.03	0.28	170	0.00%	0.00%
	IT243108	-0.03	0.24	29	0.00%	0.00%
	IT65	-0.06	0.24	36	0.00%	0.00%
	IT 553792	-0.09	0.27	18	5.56%	0.00%
Density (g/cm^3)	IT553075	0.000	0.008	170	2.94%	0.59%
	IT243108	0.002	0.008	29	0.00%	0.00%
	IT65	-0.001	0.007	36	8.33%	5.56%
	IT 553792	0.000	0.008	18	11.11%	0.00%
Average					**4.11%**	**2.00%**

Original NIRS vs. Reference Chemistry

The summary of original NIRS versus reference chemistry from 2005 is shown on Table 22. The R^2 for soybeans protein and oil are high for IT 1229 243108 and IT 1225 0065, meaning the NIRS prediction and reference chemistry agree each other. However, IT 1229 553705 seems to have a problem. It has low R^2 and high standard error. The charts in figures 13 and 14 clearly show that this instrument has some data that are not within tolerance limits. For corn, IT 1229 553075 and IT 1225 0065 are working accurately with high R^2 and low standard error. IT 1229 243108 has low R^2 and high standard error. Since IT 1229 553705 and IT 1229 243108 have problem only on one commodity (corn or soybeans), the cause of the problem may come from inaccurate reference chemistry values. Figure 12 to 16 demonstrate the working accuracy of NIRS and reference chemistry with the tolerances are y= x ± 2 SEP. The SEP values for Infratec instruments were available in tables 2 and 3, Chapter 3. By using these charts, some out of control data can be detected. The summary of original NIR versus reference chemistry and their control charts in 2004 can be seen in Appendix D.

Table 22. Original NIR vs. Reference Chemistry Summary, 2005

Constituent	Instrument	N	R^2	Standard Error	Out of Control
Soybeans Protein	IT 1229 553705	56	0.75	2.48	17.86 %
	IT 1229 243108	23	0.91	0.42	0.00%
	IT 1225 0065	14	0.98	0.67	7.14%
Soybeans Oil	IT 1229 553705	56	0.75	1.45	12.50%
	IT 1229 243108	23	0.74	0.36	4.35%
	IT 1225 0065	14	0.98	0.32	0.00%
Corn Protein	IT 1229 243108	13	0.38	0.73	7.69%
	IT 1229 553075	28	0.98	0.16	0.00%
	IT 1229 0065	4	0.97	0.13	0.00%
Corn Oil	IT 1229 243108	13	0.54	0.46	7.69%
	IT 1229 553075	28	0.65	0.17	0.00%
	IT 1229 0065	4	0.78	0.19	0.00%
Corn Starch	IT 1229 243108	13	0.78	0.45	0.00%
	IT 1229 553075	28	0.91	0.33	0.00%
	IT 1229 0065	4	0.97	0.11	0.00%
Corn Density	IT 1229 243108	13	0.12	0.02	15.38%
	IT 1229 553075	28	0.70	0.01	0.00%
	IT 1229 0065	4	0.82	0.00	0.00%

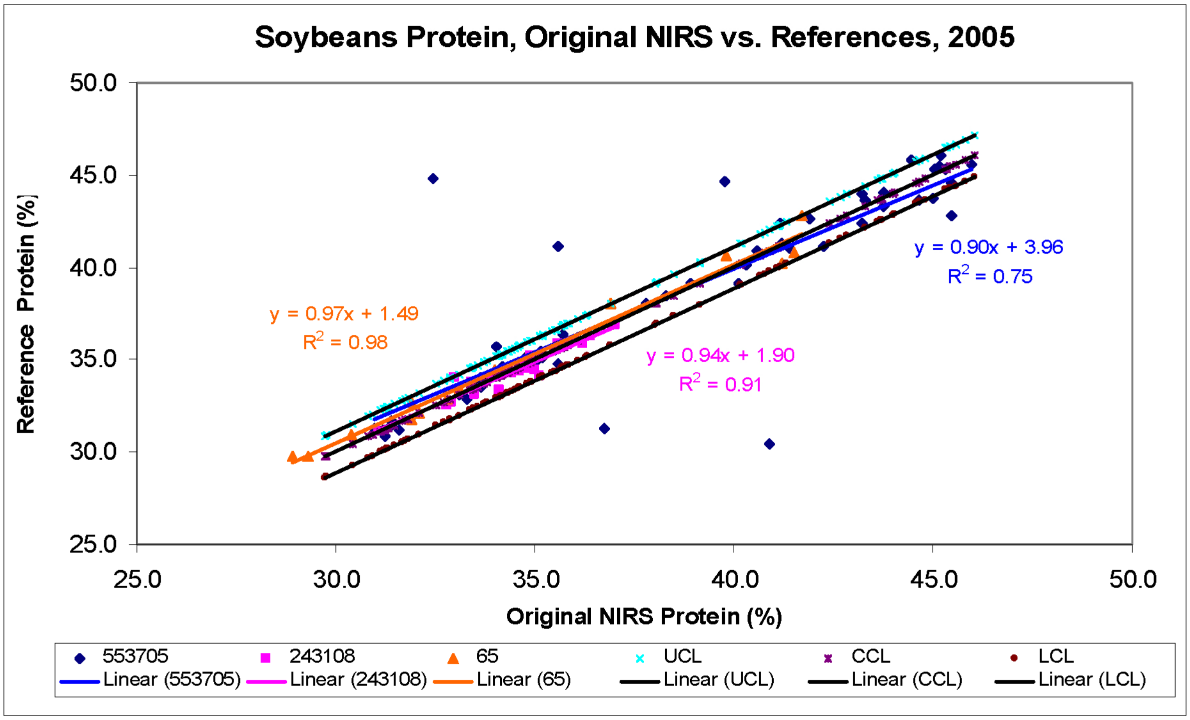


Figure 12. Soybeans Protein, Original NIRS vs. References, 2005

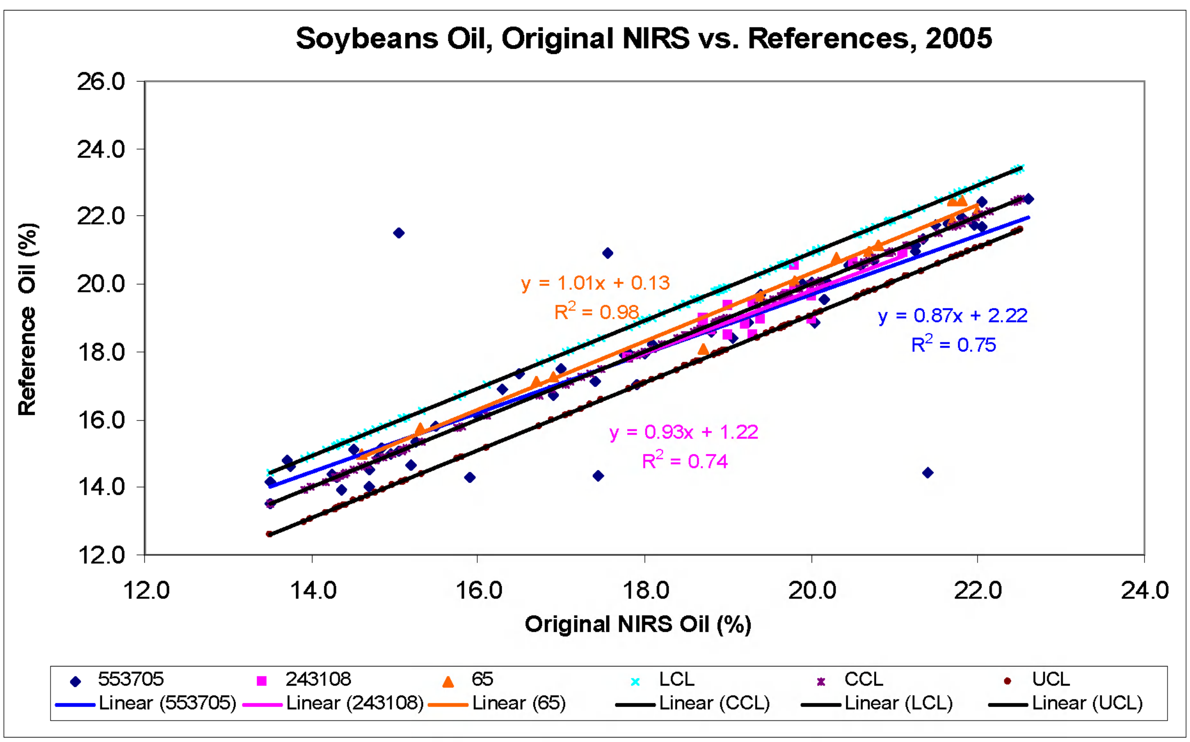


Figure 13. Soybeans Oil, Original NIRS vs. References, 2005

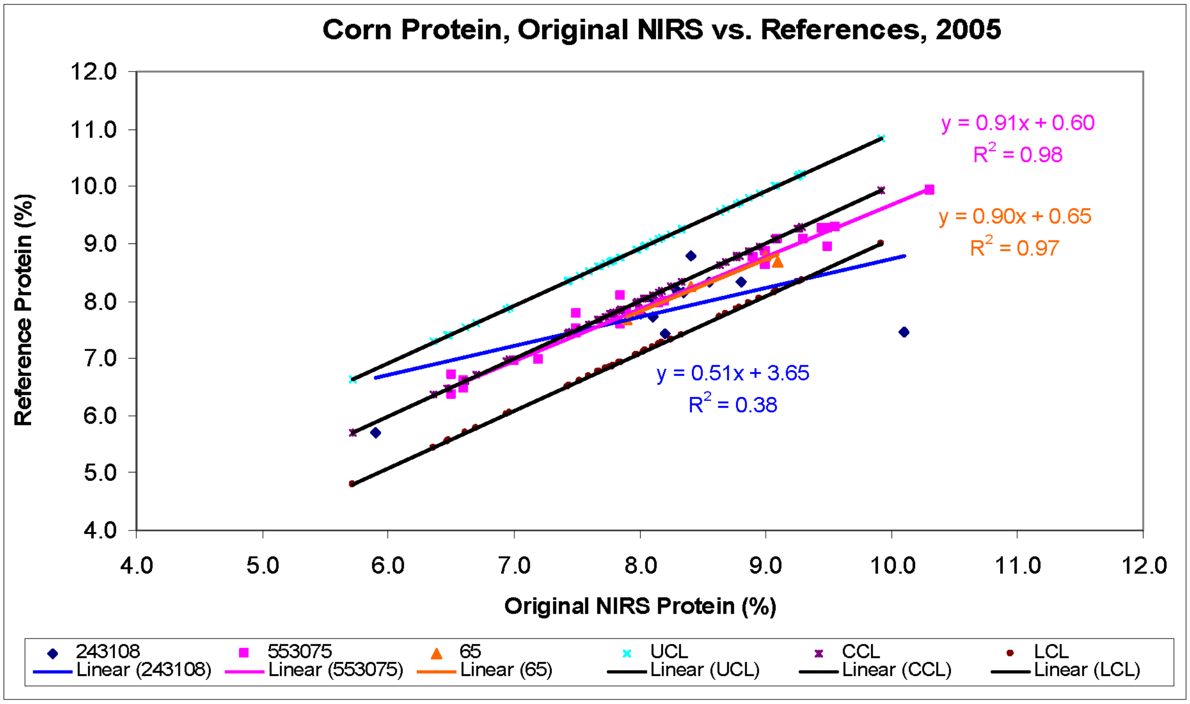


Figure 14. Corn Protein, Original NIRS vs. References, 2005

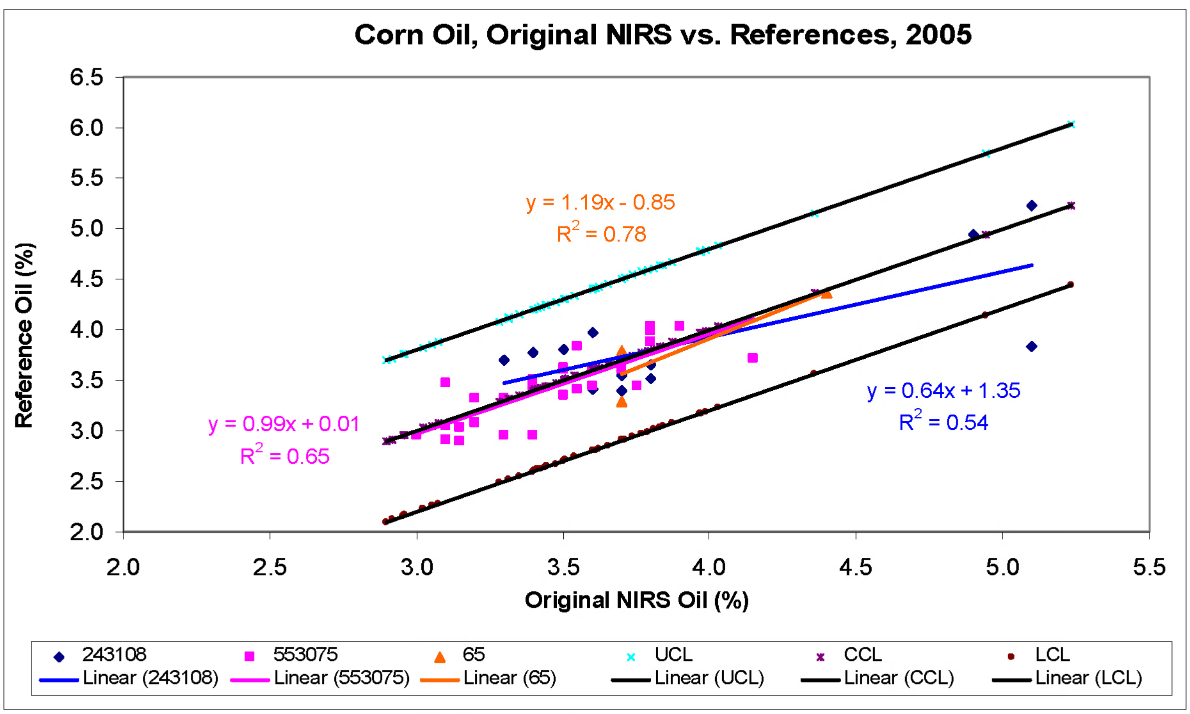


Figure 15. Corn Oil, Original NIRS vs. References, 2005

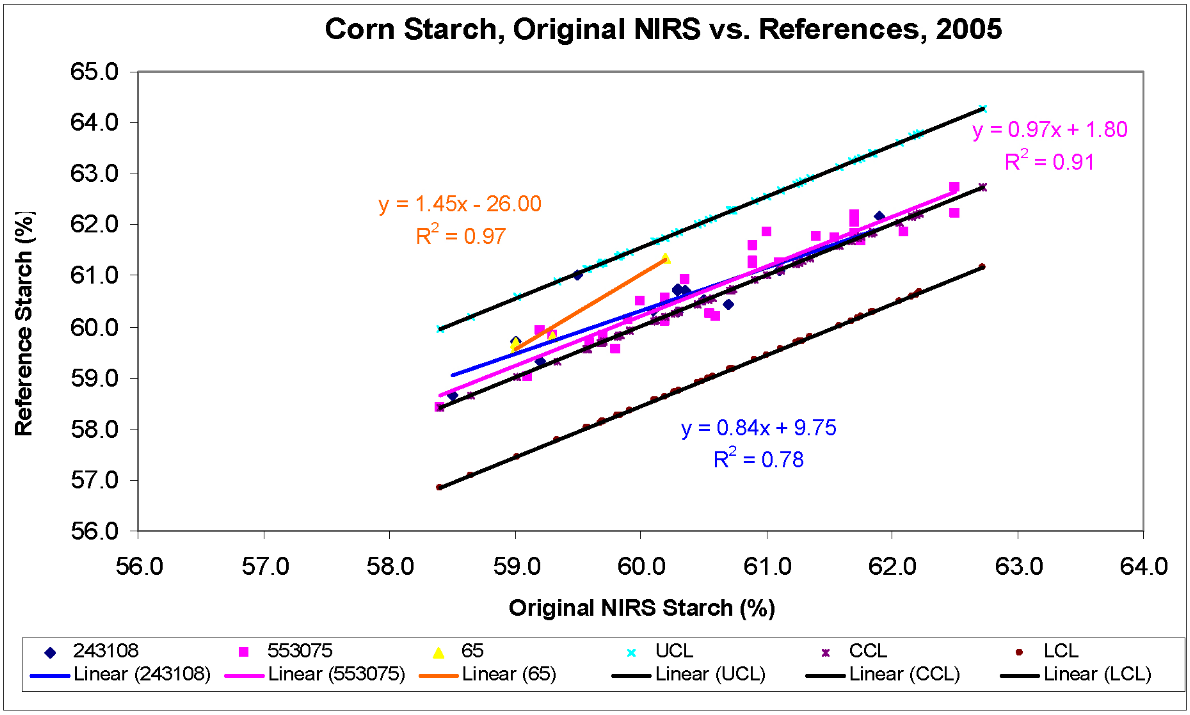


Figure 16. Corn Starch, Original NIRS vs. References, 2005

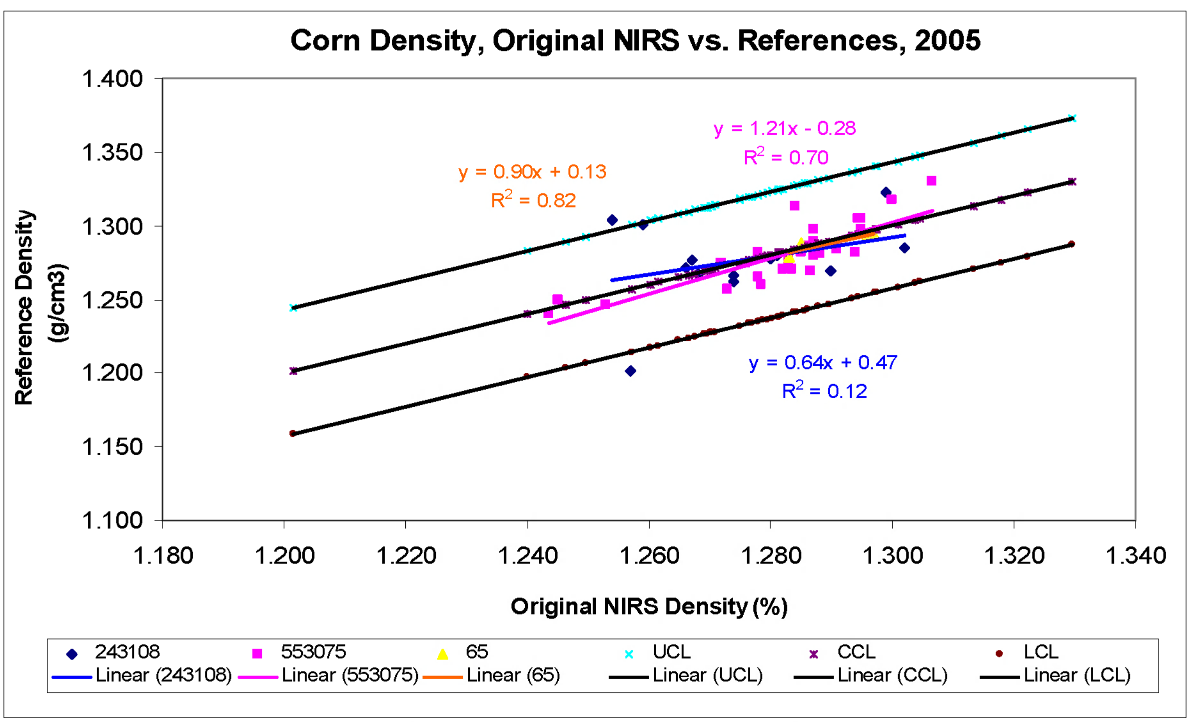


Figure 17. Corn Density, Original NIRS vs. References, 2005

Supporting Equipment

Table 23 shows the results of monthly balance check that were done from August 2004 to March 2006. The balance full scale checks that are done annually can be found in Appendix E.

Figure 18 shows the error percentage from the true value of balance monthly check. Mettler PB-153-S showed a different behavior from other balances during July to August 2005. This balance shows an increasing error and seems to have problem with accuracy. After the balance was repaired by factory technician, the balance accuracy was better. This balance is used in oven moisture; therefore the accuracy off this balance must be maintained.

Table 23. Balances Quality Control Summary, Monthly Check

Balance	Known weight (g)	N	Average of measured weight (g)	Std. dev of measured weight (g)	CV	Ave Percentage of Differences (%)	Std. Dev of Differences (%pts)
AND HR-60	20	54	20.0046	0.0052	0.03%	0.02	0.03
Denver A-250	100	54	99.9516	0.0543	0.05%	-0.05	0.05
Mettler AJ 100	50	51	50.0017	0.0028	0.01%	0.00	0.01
Mettler PB 153-S	50	54	50.0006	0.0769	0.15%	0.00	0.15
Mettler PC-4400	1000	21	998.88	0.12	0.01%	-0.11	0.01
Mettler PM- 4000	50	54	50.01	0.01	0.01%	0.01	0.01
Mettler SB-16000	1000	54	999.0	0.31	0.03%	-0.10	0.03
Seed Burro 8800	1000	54	1000.3	0.15	0.02%	0.03	0.02

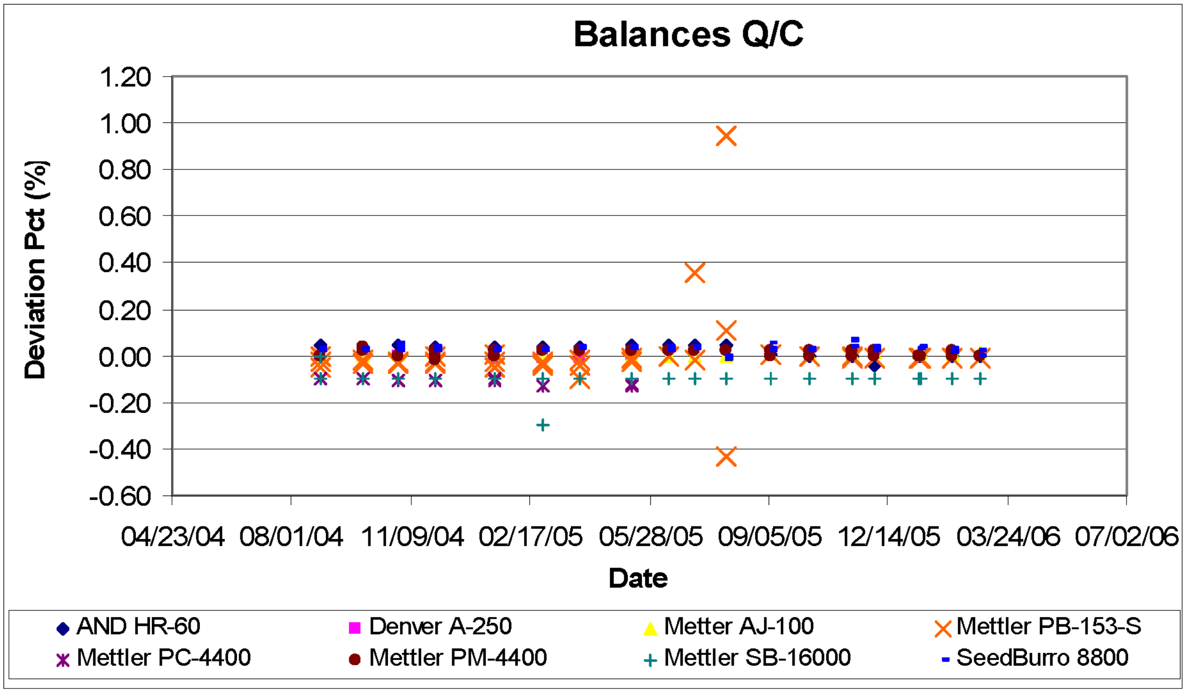


Figure 18. Balance Quality Control, Monthly Check

Table 24. IR Thermometer Quality Control Summary, Monthly Check

Thermometers		Boiling water	Room	Refrigerator
Infrared	N	58	33	33
	Average (^{0}C)	97.11	22.18	4.64
	Standard deviation (^{0}C)	1.30	1.21	1.35
Mercury	N	58	33	33
	Average (^{0}C)	99.62	22.73	4.39
	Standard deviation (^{0}C)	0.56	0.92	0.73
Differences	N	16	11	11
	Average (^{0}C)	-2.51	-0.55	0.24
	Standard deviation (^{0}C pts)	1.53	1.49	1.20
	Out of control at 95% CL	68.75%	18.18%	18.18%

Table 24 is the summary of the thermometer quality control that was done from August 2004 to January 2006. Figure 19 is the control chart of differences between Infrared and Mercury thermometer. Many out of control data came from boiling water temperature. Infrared thermometer seems to have accuracy problem in measuring high temperatures (boiling water).

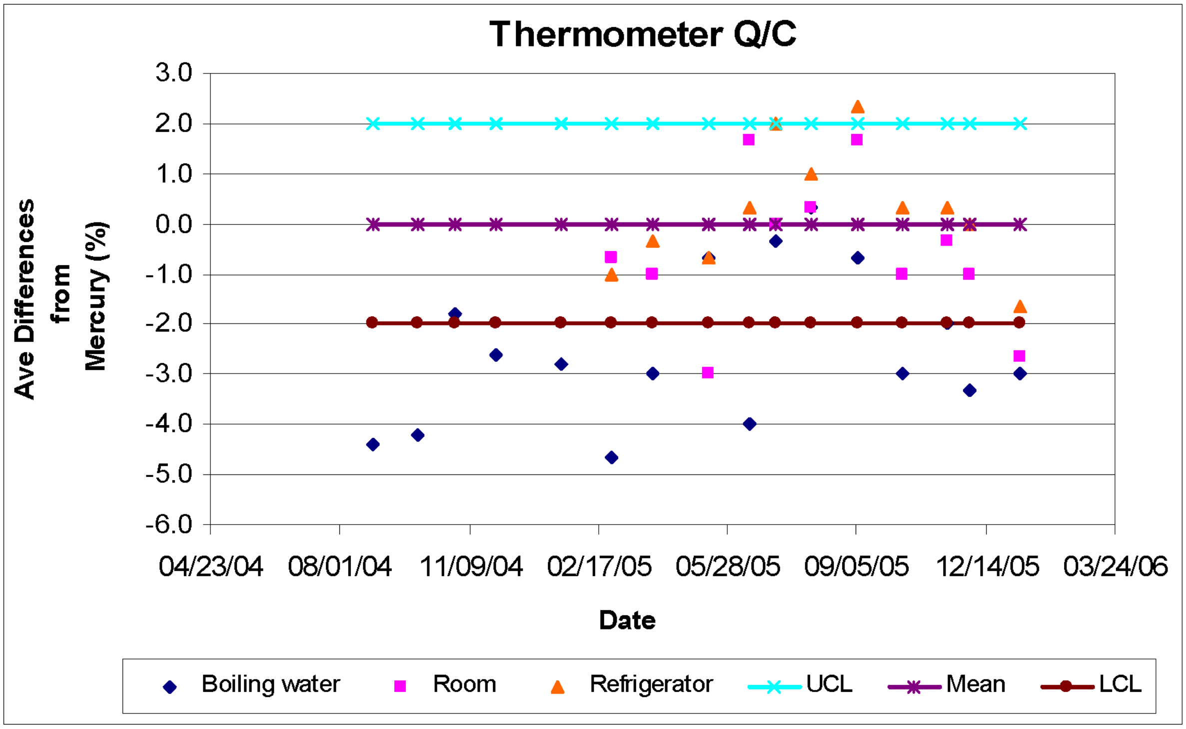


Figure 19. IR Thermometer Control Chart, Monthly Check

The Rotary and Boerner divider monthly checks are in Table 25 and 26, respectively. The Rotary divider data was taken from September 2004 to March 2006. The Boener divider data was taken from September 2005 to March 2006. The shewhart control chart of differences from true portion for each divider can be seen in Figures 20 and 21. The 40% portion of Rotary divider has out of control data at action limits. The Boerner divider is quite accurate.

Table 25. Rotary Divider Quality Control, Monthly Check

Actual Portion (%)	N	Ave of Measured Portion (%)	Std. Dev of Measured Portion (%)	CV	Ave of Diff. from Actual Portion (%)	Out of control	
						95%	99%
10	27	9.73	0.22	2.29%	0.26	11.11%	0.00%
20	27	19.94	0.30	1.49%	0.05	3.70%	0.00%
30	27	30.12	0.26	0.86%	-0.14	0.00%	0.00%
40	27	40.15	0.37	0.91%	-0.13	14.81%	7.41%

Table 26. Boerner Divider Quality Control, Weekly Check

Actual Portion (%)	N	Ave of Measured Portion (%)	Std. Dev of Measured Portion (%)	CV	Ave of Diff. from Actual Portion (%)	Out of control	
						95%	99%
50 A	7	50.37	0.60	1.19%	-0.36	0.00%	0.00%
50 B	7	49.61	0.63	1.26%	0.39	14.29%	0.00%

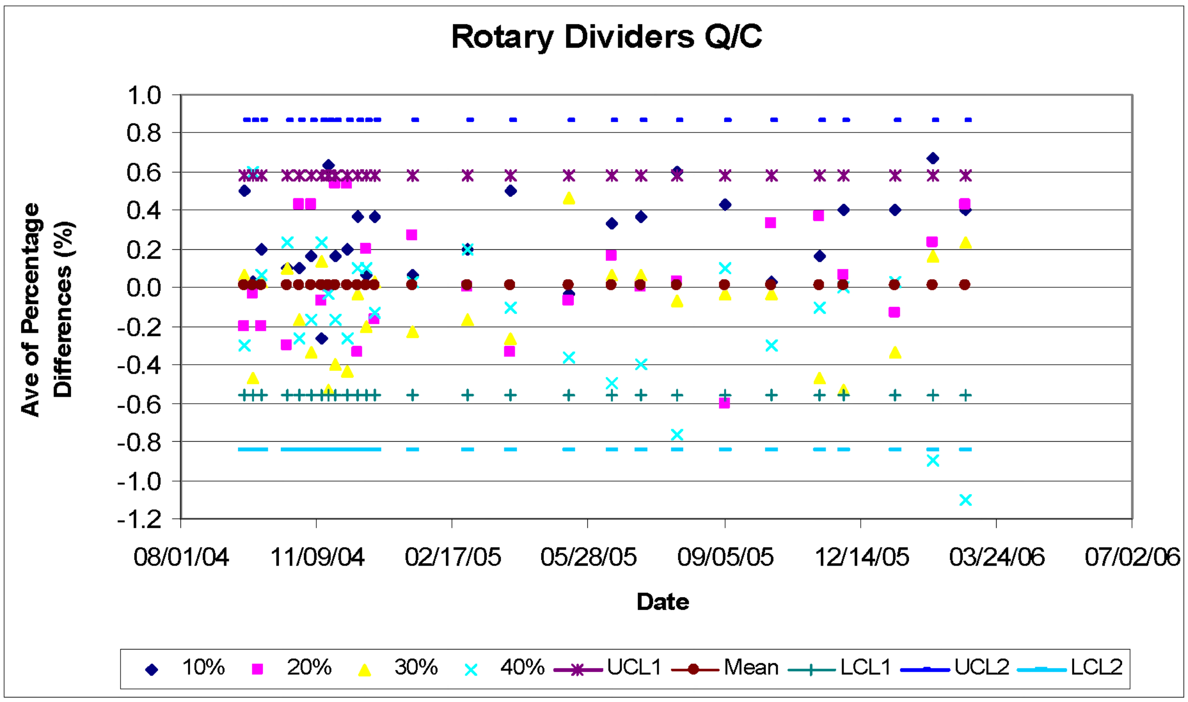


Figure 20. Rotary Dividers Control Chart, Monthly Check

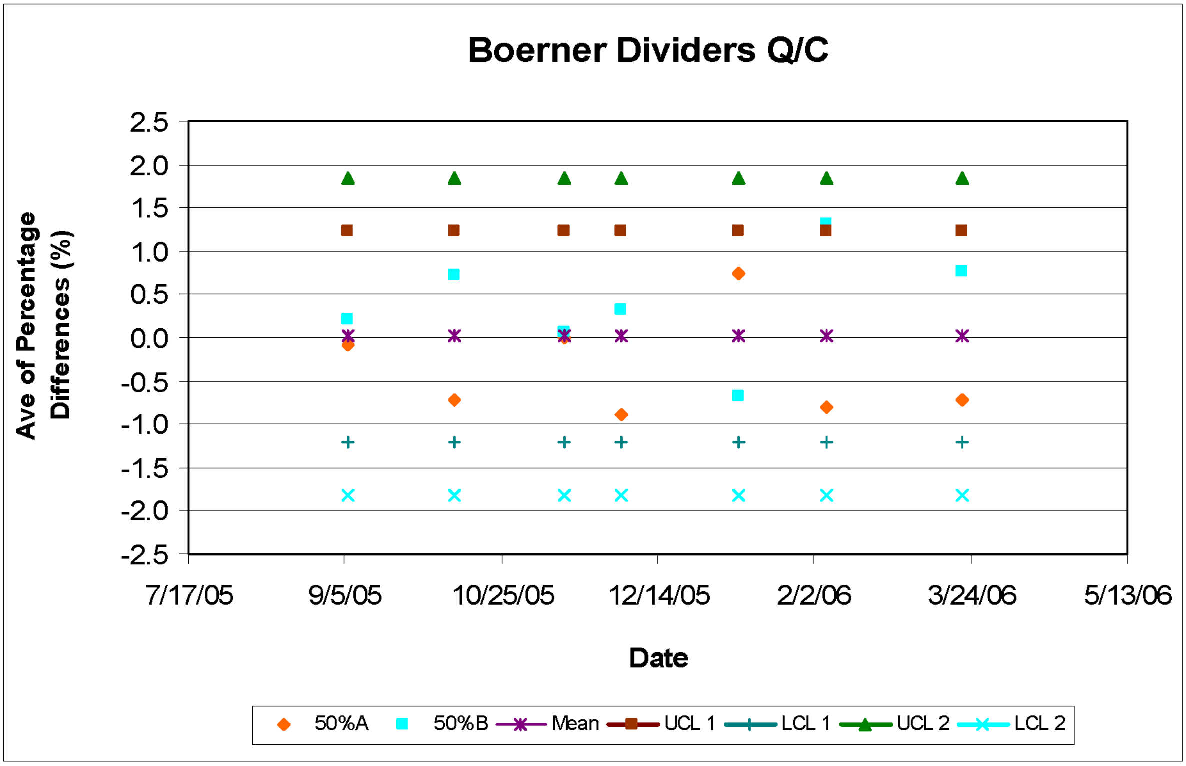


Figure 21. Boerner Dividers Control Chart, Monthly Check

Table 27 contains the results of seed counter weekly check using 300 seeds of corn and soybeans. The seed counter data was taken from February 2005 to March 2006. The seed counter has a better accuracy when it is used to count corn seeds. Figures 22 and 23 show the 95% and 99% confidence level shewhart control chart of error percentage of seed counter on 300 seeds for corn and soybeans, respectively. The soybeans control limit is bigger than the corn control limits.

Table 27. Seed Counter Quality Control Summary, Weekly Check (300 seeds)

Item		N	Average	Standard Deviation	CV	Out of control	
						95%	99%
Corn	Measured	35	300.12	0.60	0.20%		
	Pct Diff. from known	35	0.10%	0.18%		2.86%	2.86%
Soybeans	Measured	35	298.15	1.26	0.42%		
	Pct Diff. from known	35	0.64%	0.38%		2.86%	0.00%

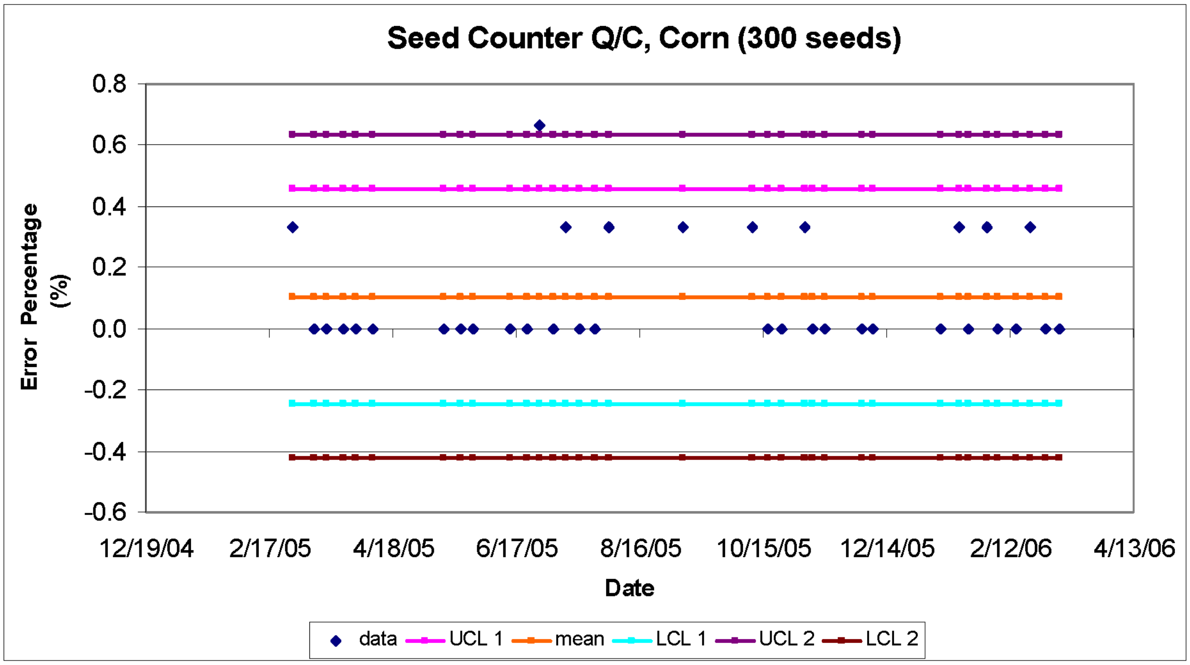


Figure 22. Seed Counter Control Chart, 300 Corn Seeds, Weekly Check

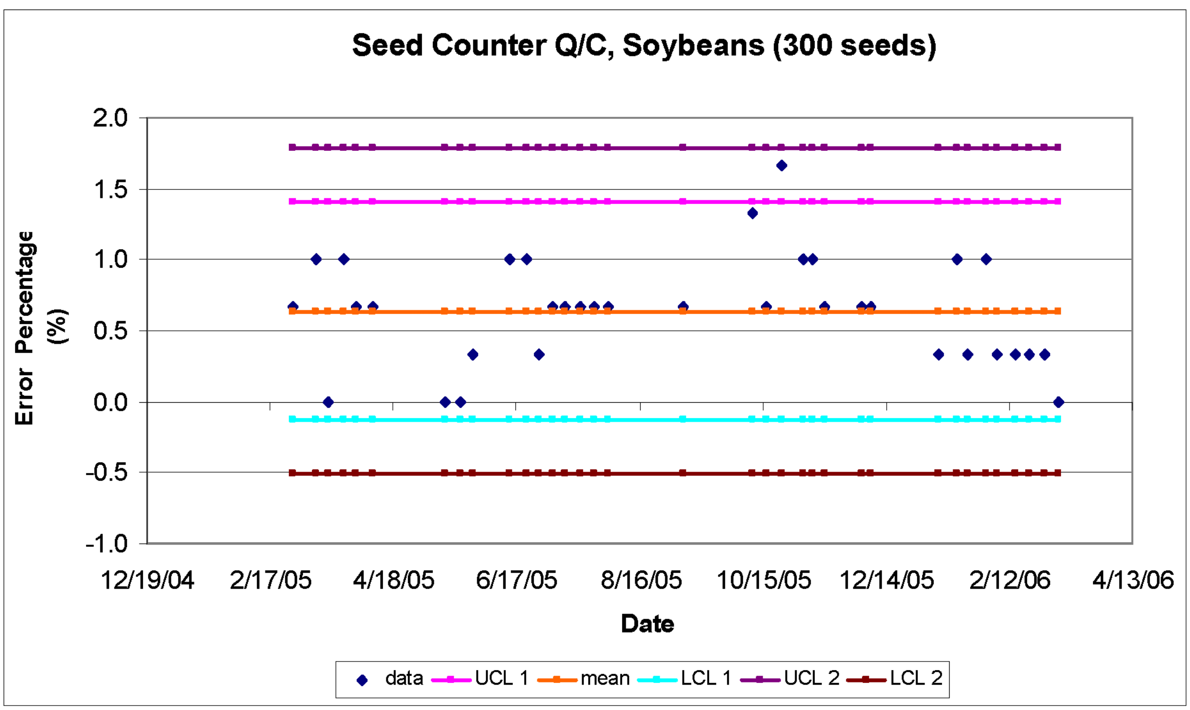


Figure 23. Seed Counter Control Chart, 300 Soybeans Seeds, Weekly Check

Table 28 contains test weight results done in kettle and GAC instruments. The data was taken weekly from November 2004 to March 2006 using corn sample 20040049. The test weight values were corrected using equation 6 in the previous chapter. On average kettle and GAC test weight have similar results. However, standard deviation between reps on GAC 2100 is quite large compared to the others. From Figures 24 and 25, test weight increases over time for both kettle and GAC test. Figure 26 shows the moisture of corn sample decreases over time. Figure 27 shows the 95% confidence level shewhart control chart of test weight differences between quart/pint and GAC 2000/2100. There are 4 out of control data over 43 data points (9.30%). Figure 28 plots the average test weight of quart and pint versus the average test weight of GAC 2000 and 2100. This chart indicates that operator training is needed for the kettle test weight.

Table 28. Test Weight Quality Control Summary, Corn Sample 20040049, Weekly Check

	N	Average (lb/bu)	Std. Dev (lb/bu)	CV	Std. Dev between reps (lb/bu)
Quart	43	58.08	0.48	0.83%	0.25
Pint	43	57.69	0.59	1.02%	0.28
GAC 2000	43	57.82	0.47	0.81%	0.29
GAC 2100	43	57.98	0.55	0.96%	0.45
Diff. between Quart and Pint	43	0.39	0.54		
Diff. between GAC 2000 and 2100	43	-0.16	0.51		
Diff. between Quart/Pint and GAC 2000/2100	43	-0.02	0.49		

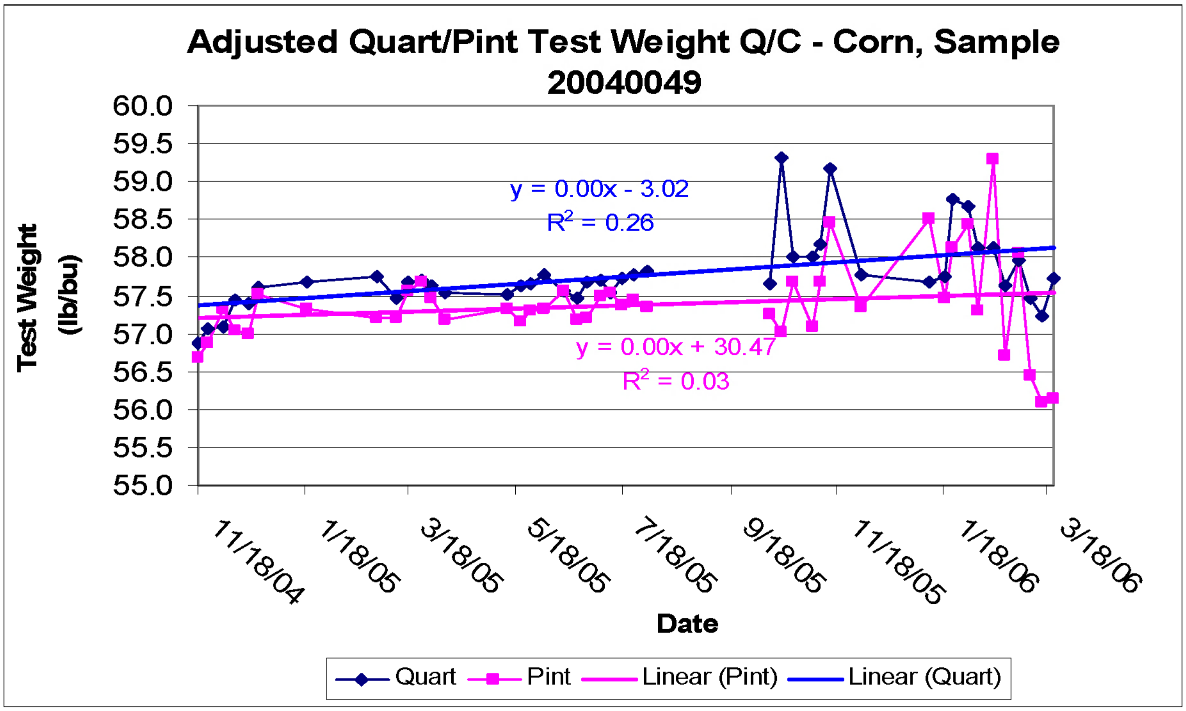


Figure 24. Adjusted Quart/Pint Test Weight Quality Control Chart, Corn Sample 20040049

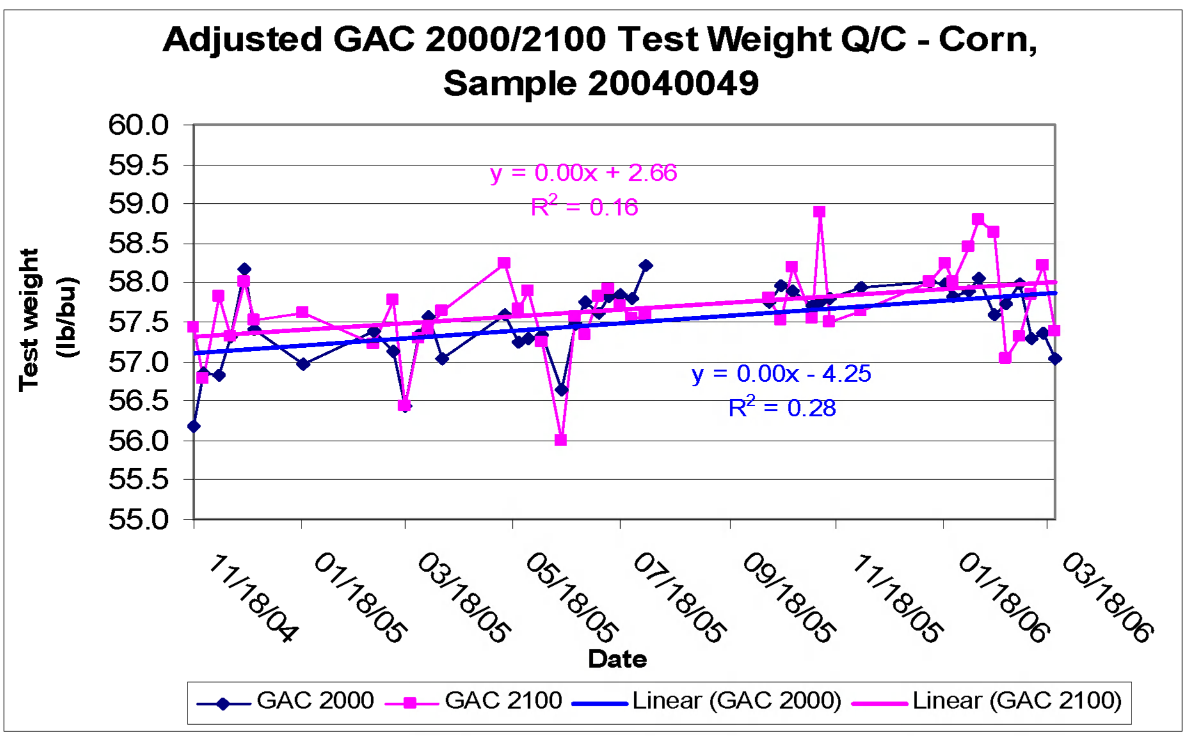


Figure 25. Adjusted GAC 2000/2100 Test Weight Quality Control Chart, Corn Sample 20040049

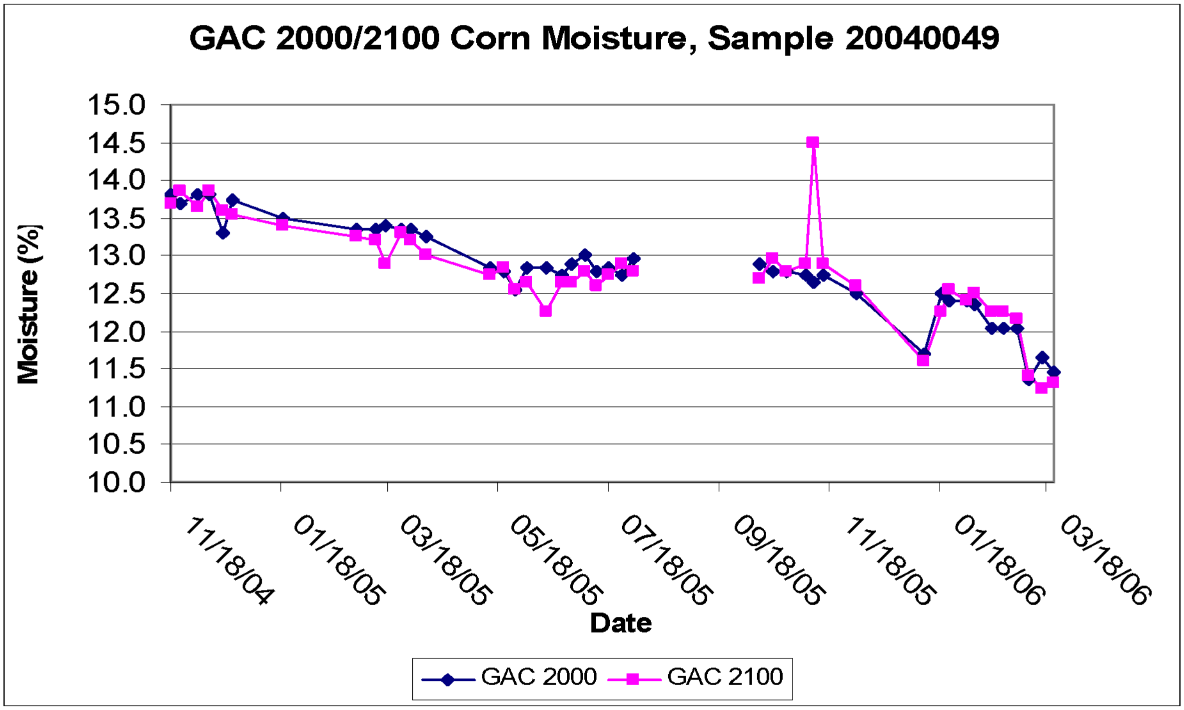


Figure 26. GAC 2000/2100 Corn Moisture Weekly Check Chart, Sample 20040049

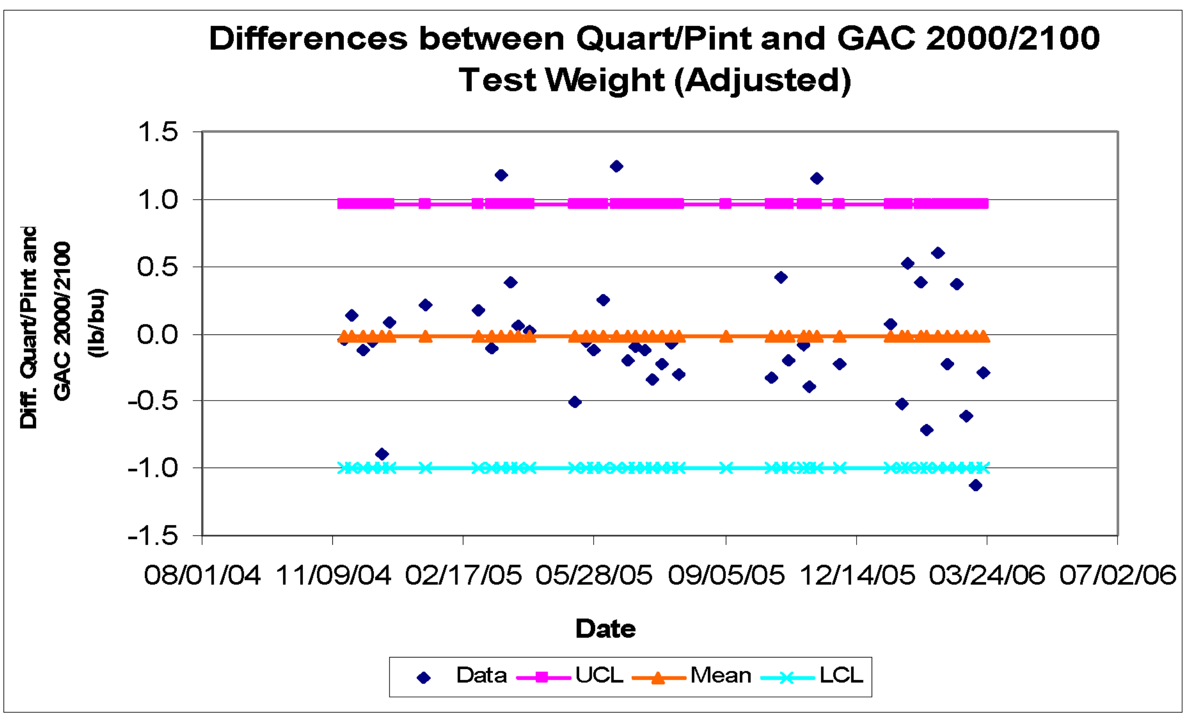


Figure 27. Differences between Quart/Pint and GAC 2000/2100 Test Weight Control Chart, Corn Sample 20040049 (Adjusted).

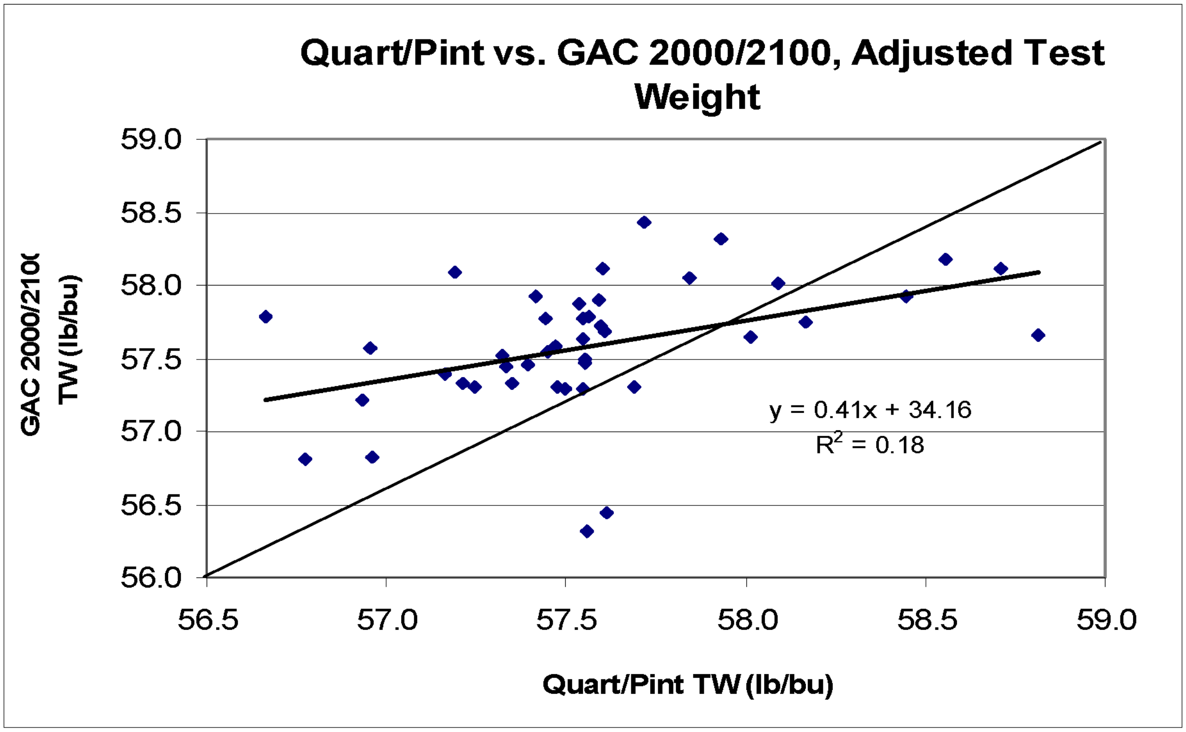


Figure 28. Test Weight GAC 2000/2100 vs. Quart/Pint, Corn Sample 20040049 (Adjusted)

The result of water test weight yearly check is shown in table 29. The data was taken in 2003, 2004 and 2005 for both quart and pint. The water test weight was done twice in 2003 and 2004. The water test weight on pint has a slightly bigger coefficient of variability than quart. Figure 29 and 30 plot the differences between measurement and actual weight of yearly water test weight on quart and pint respectively with ±1 gram as control limits.

Table 29. Water Test Weight Quality Control Summary, Yearly Check

	Quart	Differences	Pint	Differences
N	5	5	5	5
Standard weight	1098.10 g		549.05 g	
Average	1100.00 g	1.88 g pts	550.40 g	1.36 g pts
Standard Deviation	5.85 g pts	5.85 g pts	1.55 g pts	1.55 g pts
CV	0.17%		0.25%	

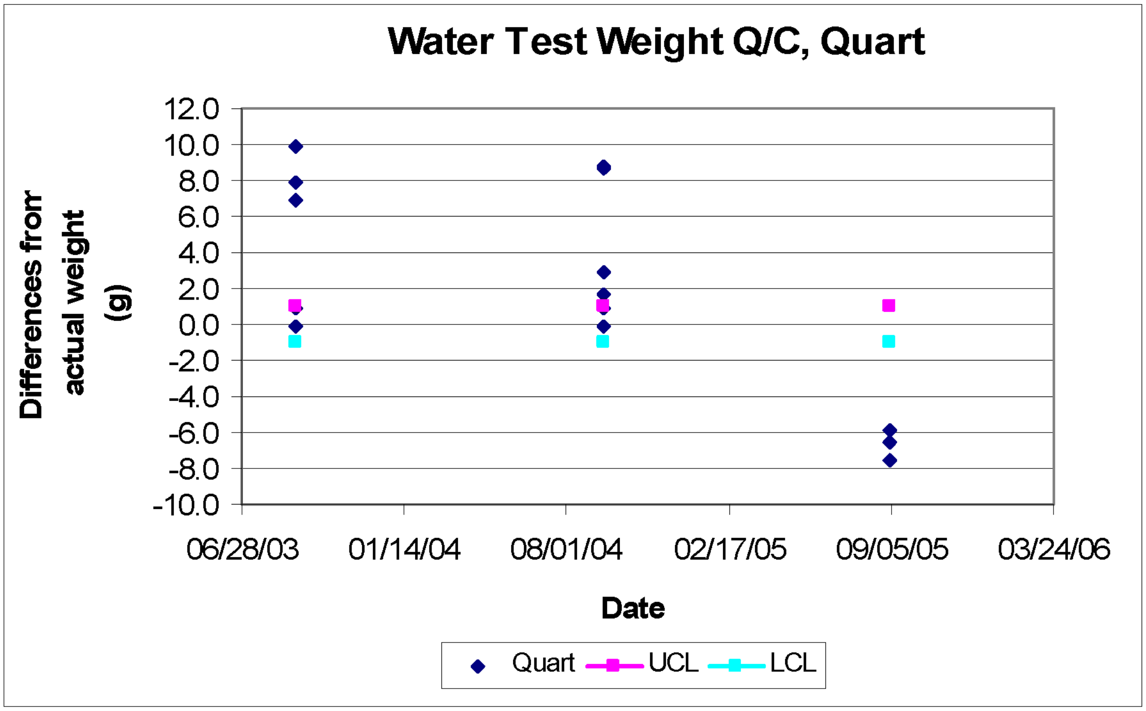


Figure 29. Water Test Weight Chart, Quart, Yearly Check

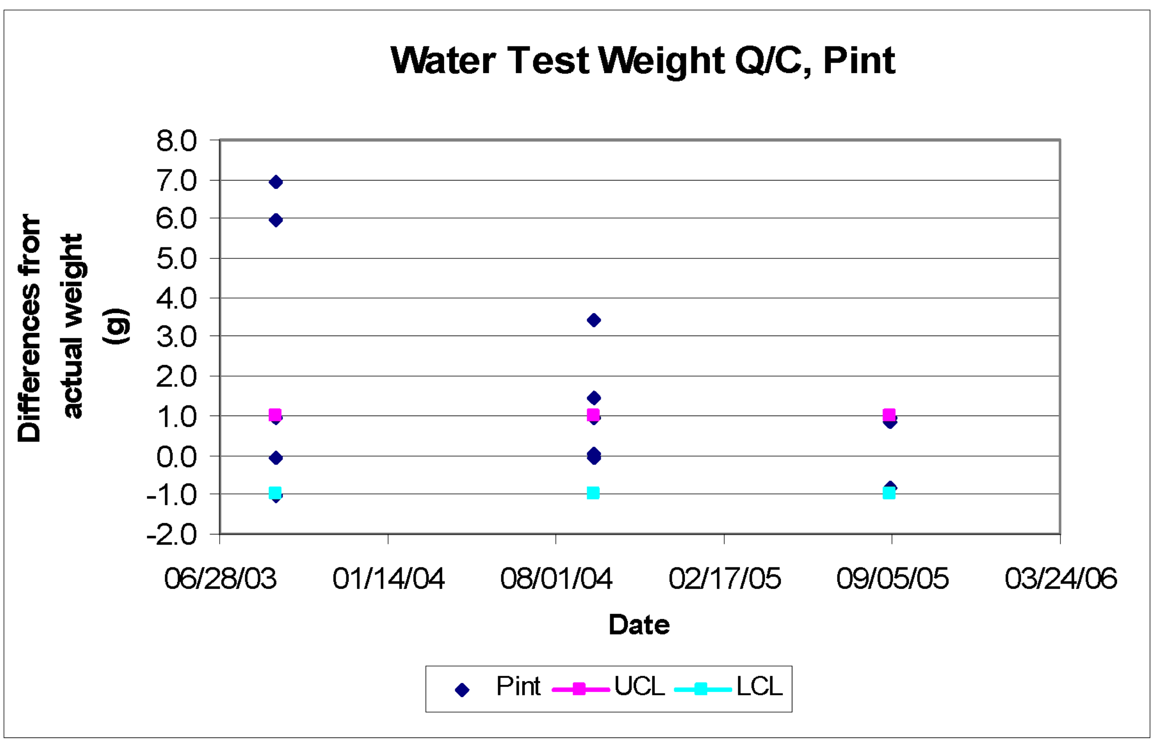


Figure 30. Water Test Weight Chart, Pint, Yearly Check

Laboratory Air Room Conditions

Figure 31, 32 and 33 show the examples of monthly chart that plots climate data in the ISU-GQL. The data for these charts were taken in February 2006. Relative Humidity varied from 21 to 27%. Temperature ranged from 21.5^{0}C – 23.5^{0}C which is quite consistent and atmospheric pressure ranged from 965-1010HPa.

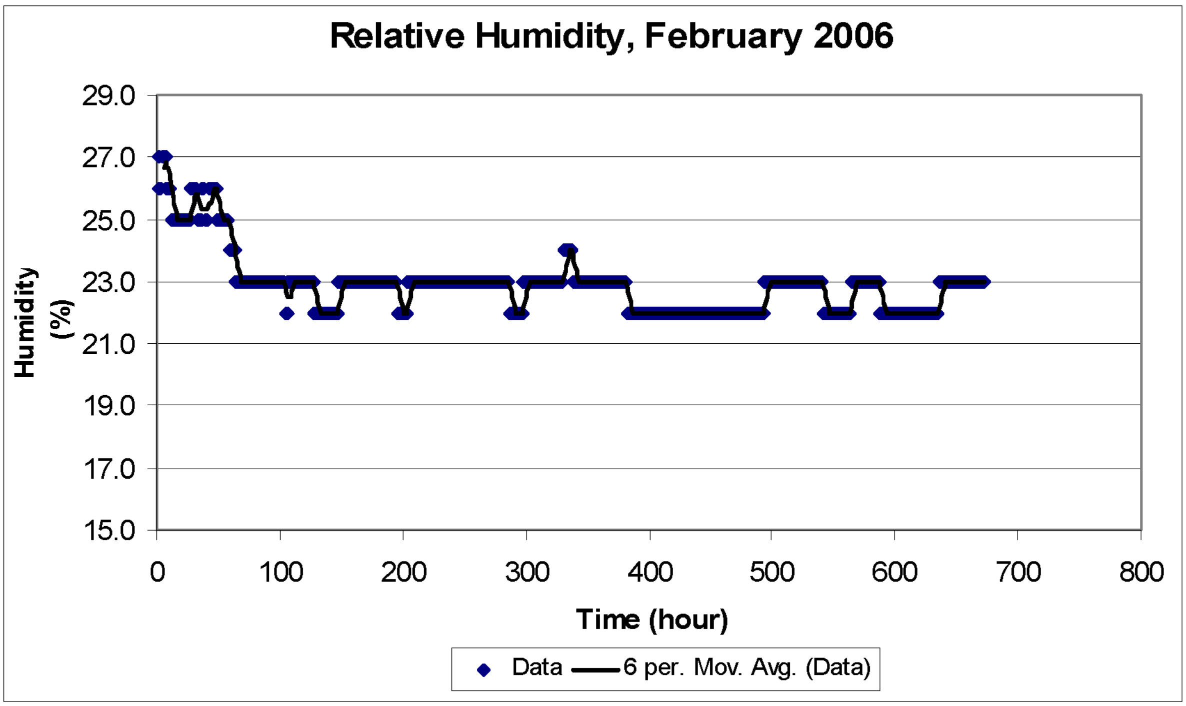


Figure 31. Laboratory Humidity in February 2006

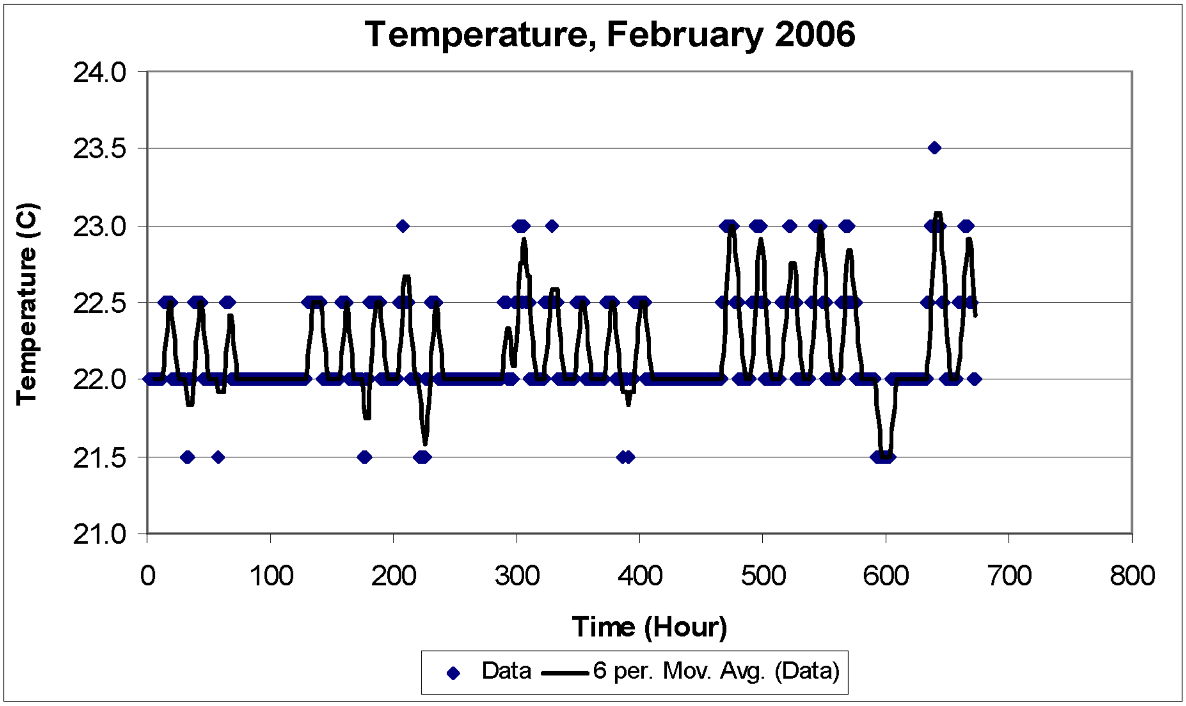


Figure 32. Laboratory Temperature in February 2006

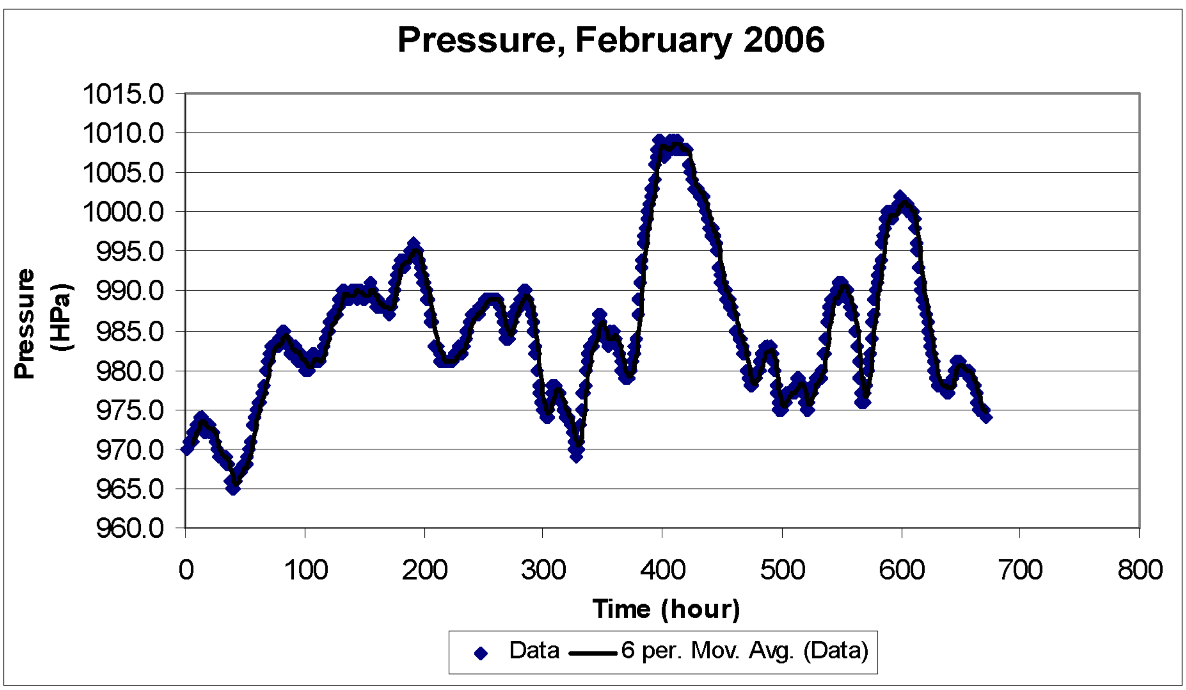


Figure 33. Laboratory Pressure in February 2006

CHAPTER 6. QUALITY CONTROL PROGRAM FOR THE REFERENCE DATA

The reference chemistry data in ISU-GQL consist of internal lab data and external lab data. The internal lab data are the analytical data from the measurements done by ISU GQL. The data include oven moisture (corn and soybeans) and corn density data. The external data are the analytical data that ISU GQL receives from other labs. The data include proximate analysis (protein, oil, starch, fiber), amino acids and fatty acids. Since the documentation for the reference data were poorly organized, a better documentation was developed.

ISU-GQL used Microsoft Access database as a solution for data management. Five data tables were constructed for each corn and soybeans database. The structure and content of the soybeans and corn reference chemistry database are shown in tables 30 and 31, respectively. The Master table contains general information of samples. The proximate table has information about reference values from chemistry lab. Moisture and Physical properties table contains moisture measurement and test weight results at first scanning. ID3 is the eight digit identification number starting with 4 digits of year (e.g. 20050019) that is used to link the tables. Table 32 contains information about laboratories that do reference chemistry, the method used and their units. The procedures to update and to use Access database are documented in the Appendix J.

Table 30. Soybeans Database Structure and Content

Master	Proximate	Moisture and Physical Properties	Fatty Acid	Amino Acid	
ID3	ID3	ID3	ID3	ID3	VAL[a]
ID1	Date of the test	Date of the test	Universal ID	ID1	MET[a]
Universal ID	Lab doing the test	Lab Doing the test	Date of test	Date of the test	ISO[a]
State	Protein[a]	GAC MC	Lab doing the test	Lab doing the test	LEU[a]
Origin	Protein Q/C Bias[a]	Oven Moisture 1	On hand date	TAU[a]	TYR[a]
Variety Company	Oil[a]	Std. Dev Reps 1	Palmitic[a] 16:0	HYP[a]	PHE[a]
Variety No	Oil Q/C Bias[a]	Oven Moisture 2	Stearic[a] 18:0	ASP [a]	HYL[a]
Class	Fiber[a]	Std. Dev Reps 2	Oleic[a] 18:1	THR [a]	HIS[a]
Original Weight	Fiber Q/C Bias[a]	Test Weight kettle	Linoleic[a] 18:2	SER[a]	ORN[a]
Current Weight	NSI[a]	Thousand Seeds Weight[a]	Linolenic[a] 18:3	GLU[a]	LYS[a]
Date of Updated weight			Total Saturates[a]	PRO[a]	ARG[a]
Proximate Flag				LAN[a]	TRY[a]
AA flag				GLY[a]	TOTAL[a]
FA flag				ALA[a]	
Sugars flag				CYS[a]	

[a]13% moisture basis

Table 31. Corn Database Structure and Content

Master	Proximate	Moisture and Physical Properties	Density	Amino Acid	
ID3	ID3	ID3	ID3	ID3	CYS[a]
ID1	ID1	Date of test	Date of test	ID1	VAL[a]
Universal ID	Date of test	Lab doing the test	Lab doing the test	Date of test	MET[a]
State	Lab doing the test	GAC MC	Density[a]	Lab doing the test	ISO[a]
Origin	Protein[a]	Oven Moisture 1		TAU[a]	LEU[a]
Variety Company	Protein Q/C bias[a]	Std. Dev Reps 1		HYP[a]	TYR[a]
Variety No	Oil[a]	Oven Moisture 2		ASP[a]	PHE[a]
Class	Oil Q/C bias[a]	Std. Dev Reps 2		THR[a]	HYL[a]
Original Weight	Starch[a]	Test Weight kettle		SER[a]	HIS[a]
Current Weight	Starch Q/C bias[a]	Test Weight[a]		GLU[a]	ORN[a]
Date of Updated weight		Thousand Seeds Weight[a]		PRO[a]	LYS[a]
Proximate flag				LAN[a]	ARG[a]
AA flag				GLY[a]	TRY[a]
				ALA[a]	TOTAL[a]

[a]15% moisture basis

Table 32. Corn and Soybeans Constituent Methods

Constituent	Reference Laboratory	Method	Units
Corn			
Moisture	Iowa State University - GQL	AACC 44-15A	% wt
Moisture Std. Dev			% pts
Protein	Eurofins, Des Moines, Iowa	AOAC 990.03	% wt
Oil	Eurofins, Des Moines, Iowa	AOAC 920.39	% wt
Calculated Starch[a]	Iowa State University - GQL	Calculated to A-20 Corn Refiners Association	% wt
Starch	Eurofins, Des Moines, Iowa	A-20 Corn Refiners Association	% wt
Density	Iowa State University - GQL	AccuPyc 1330 Pyconometer Operator's Manual V2.01	g/cc
Amino Acids	University of MO.	AOAC 982.30	% wt
Test weight	Iowa State University - GQL	Kettle, GAC instrument	lb/bu
Weight	Iowa State University - GQL		gram
Soybeans			
Moisture	Iowa State University - GQL	AOCS Ac 2-41	% wt
Moisture Std. Dev			% pts
Protein	Eurofins, Des Moines, Iowa	AOAC 990.03	% wt
Oil	Eurofins, Des Moines, Iowa	AOCS Ac 3-44	% wt
Calculated Fiber[b]	Iowa State University - GQL	Calculated to AOCS Ba 6-84	% wt
Fiber	Eurofins, Des Moines, Iowa	AOCS Ba 6-84	% wt
Fatty Acids	Iowa State University - Fehr	GC method by Hammond	% of oil
Amino Acids	University of MO.	AOAC 982.30	% wt
NSI	Eurofins, Des Moines, Iowa	AOCS ba 11-65	% of Protein
Test weight	Iowa State University - GQL	Kettle, GAC instrument	lb/bu
Weight	Iowa State University - GQL		gram

[a]60.99-0.9175*protein-1.110*oil+8.58*density
[b]11.09-0.117*(protein+oil)

The quality control program for reference data done by ISU-GQL are explained below:

Oven Moisture

Oven moisture data are collected every year when ISU-GQL validates calibrations for NIRS instruments. Every sample is divided into three replicates. The moisture final results are the average of three replicates. Ten percent of the samples are duplicated.

There are three charts built for quality control purposes:

- Chart of average moisture from three replicates versus averages of difference from each pair of replicates for the dishes and duplicates. This chart demonstrates the precision of oven moisture.
- Chart of consecutive number versus the standard deviation from three replicates of dishes and duplicates. This chart is built to know the samples repeatability.
- Control chart of differences between average moisture of dishes and duplicates. This chart is created to know the precision of oven moisture over many samples and over time.

Tolerance determination for oven moisture follows the USDA-GIPSA method. USDA-GIPSA method states that the difference between replicate analyses should be as follows:

$\leq$ 0.25% for corn containing $\leq$15% moisture,

$\leq$ 0.30% for corn containing > 15% moisture, and

$\leq$ 0.20% for all soybeans.

(USDA-GIPSA, 1998)

The tacit assumption is that the above tolerances are 2 standard deviation.

The data and quality control chart of oven moisture are documented in spreadsheet by year. The averages of oven moisture and their duplicates from year to year are saved in the "Moisture and Physical Properties" table of the corn or soybeans Access database. The others information such as standard deviation from oven moisture replicates, average moistures from GAC instruments, test weight from kettle and GAC instruments, and 1000 seed weight at 15% and 13% moisture basis for corn and soybeans, respectively are also saved in this table.

Corn Density

The corn density data are also collected annually to update the NIRS calibration. To correct density for moisture, sample moisture is measured three times each using GAC 2000 and GAC 2100. Average and standard deviation of six measurements are calculated. This moisture averages is used to determine constant moisture basis for density. Density is measured three times. The averages of corn density from the three measurements are converted into 15% moisture basis using the equation developed by Redding et. al (1991):

Density (15%) = average density – 0.00289*(15% - average moisture basis) (eq. 7)

A chart that plots the standard deviations of density describes the precision of measurements. The corn density yearly calibration data are documented in the spreadsheet by year. This data then is moved to the “Density 15% “table in the Microsoft Access database.

A quality control process for the pyconometer reproducibility was also developed. The pyconometer is checked daily during the NIR yearly calibration using one corn sample. The same sample is also used to check two GAC instruments. The sample is changed at least once a year. The 95% shewhart control chart is built for the pyconometer quality control. So it uses its own data. The data and control chart are stored in the spreadsheet.

Instrument standardization sample set are also checked as part of reference density quality control. Standardization samples are the samples that are used to standardize the same brand of instruments after the calibration is done. Standardization samples also have function as checked/repeated samples. All the results from standardization samples set are documented in the spreadsheet and Access databases. The calculations are done in the spreadsheet. The differences between new results and average of first three measurements of repeated samples are calculated. The average of first three measurements is fixed for future test results evaluation. However, the differences between each pair of these first three measurements have to be within the tolerance limits. The calculation for setting the tolerance limits will be shown in proximate analysis section since the same method is used. A chart of number of measurements versus standard deviation of repeated samples is built in every yearly standardization spreadsheet. This chart is constructed to know the consistency of measurements done by lab. A control chart of differences between every measurement and average of first three measurements with fixed tolerance limits is also created in the yearly standardization spreadsheet. Out of control data and/or bad samples will be identified by this chart.

Quality control program for reference data done by external labs are explained below:

Proximate Analysis

The proximate analysis data are outsourced from Eurofins Lab, Des Moines. Proximate analysis quality control is developed to ensure the quality of calibration development. When ISU-GQL submits yearly calibration samples or samples for any other purpose, repeated and blind duplicate samples are also sent. The repeated samples are usually from the instrument standardization sample set. Blind duplicate samples can be standardization or some of the yearly calibration samples. They have to be at least 5% of the total submitted samples (for both repeated and blind duplicate samples). The repeated and blind duplicate samples are blind coded so that samples could not be identified by the laboratory as a check sample or as a duplicate.

The differences between new results and the average of first three measurements of repeated samples are calculated. The average of first three measurements is fixed for the future use. It means that the next tests will be evaluated using this average. However, the differences between each pair of the first three measurements have to be within the tolerance limits. If the average of differences between new results and average of first three measurements of repeated samples are within the tolerance limit, then bias are not applied. However, the standard deviation of differences of repeated samples also has to be acceptable.

If the average of differences between the new results and the average of first three measurements of repeated samples and their standard deviation are not within the tolerance limit, then bias may need to be applied for this set of data. This value then is applied to the all submitted samples set after moisture basis are converted to 15% and 13% for corn and soybeans, respectively. The purpose of applying bias is to keep the accuracy of calibration. The flowchart of this process is in Figure 34. The process of proximate analysis and corn density tolerance setting is explained below:

Proximate and Corn Density Tolerance Setting

The tolerance limits for differences between current measurement and the average of first three measurements were calculated. The measurements considered as outliers were not used in the tolerance calculation. Outlier removal process was done for every sample using mean ± 1 standard deviation. The reason of using 68% confidence level (about 1 standard deviation from mean) is because 2 and 3 standard deviations from the mean, which correspond to 95% and 99% confidence level, respectively were not able to detect any outliers. Bad results tend to protect themselves. The tolerance limits were calculated based on average of three methods using 95% confidence level. The three methods were:

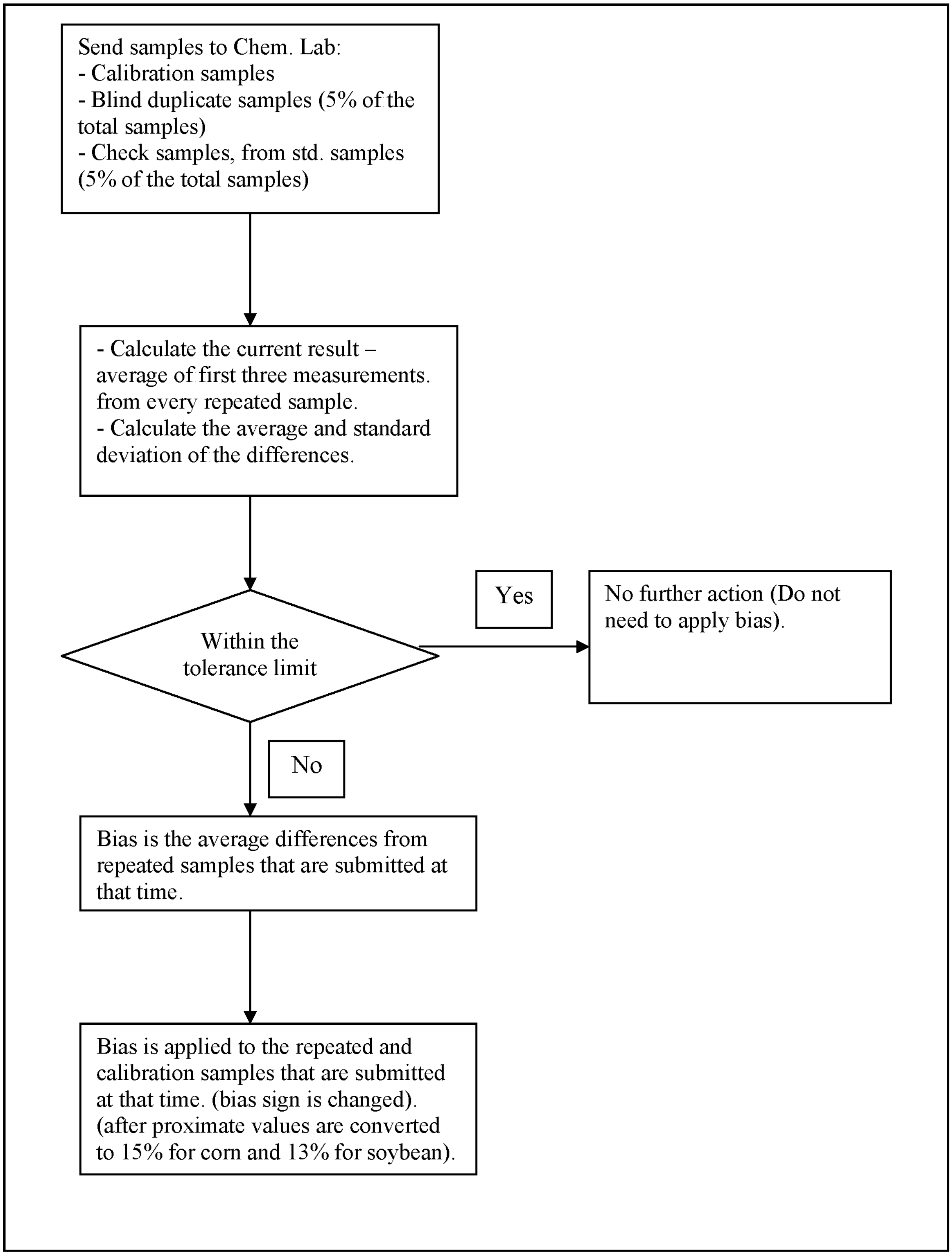


Figure 34. Proximate Analysis Quality Control Flowchart

1. The first method :

   tolerance limits = ± the average of $2 \times \sqrt{2 \times \sigma^2}$ each sample. (eq.8)

   σ^2 = variance of all measurements of each sample.

   (Mullins, 2003)

2. The second method:

   tolerance limits = ± $2 \times \sqrt{2 \times \sigma^2{}_{pooled}}$ (eq.9)

   $\sigma^2{}_{pooled}$ = the pooled variance of all samples.

   $$\sigma^2{}_{pooled} = \frac{\sigma_1{}^2 \times (n_1 - 1) + \sigma_2{}^2 \times (n_2 - 1) + \ldots + \sigma_n{}^2 \times (n_n - 1)}{(n_1 - 1) + (n_2 - 1) + \ldots + (n_n - 1)} \quad \text{(eq.10)}$$

Mullins suggested the average variance based on the assumption that the sample size each sample was the same. Since the sample size of the each sample is not the same, a pooled variance was used (Mullins, 2003), (Vaderman and Jobe, 2001).

3. The third method based on shewhart control chart theory. Because differences were taken from every measurement from mean of each sample, the mean differences were zero. The tolerance limits came from average of standard deviation of the difference times two.

   tolerance limits = ± $2 \times average\sigma_{diff}$ (eq.11)

   (Garfield et al, 2000)

Tolerance limits for the differences were calculated for protein and oil of corn and soybeans, and corn density. Tolerance limits were not calculated for corn starch and soybeans fiber, since they are calculated from other reference values. The tolerances for standard deviation of each constituent were calculated using the equation 12. Since the smaller standard deviation is better, the tolerances were only calculated for the upper limit using alpha= 0.05.

$$\text{average of } s\sqrt{\frac{n-1}{c_{1-\alpha}}} \text{ from each sample} \qquad \text{(eq. 12)}$$

n= number of measurement

s = standard deviation

c= chi-square value at 1-α (α= 0.05)

(Steel et al, 1997)

Table 33 summarizes the number of samples and measurements before and after removing outliers. Each sample has different number of measurements. The number of measurements before and after outlier removal for each sample can be seen in Appendix F.

Table 33. Number of Samples and Measurements Summary

Constituents	Number of samples	Number of measurements	
		Before	After
Soybeans Protein	30	205	147
Soybeans Oil	30	185	130
Corn Protein	39	184	120
Corn Oil	39	171	124
Corn Density	30	99	81

Table 34. Corn Proximate and Density Tolerance Limits

Method	Protein (%)		Oil (%)		Density (g/cc)	
	LCL	UCL	LCL	UCL	LCL	UCL
Method 1	-0.28	0.28	-0.23	0.23	-0.008	0.008
Method 2	-0.36	0.36	-0.38	0.38	-0.011	0.011
Method 3	-0.20	0.20	-0.16	0.16	-0.005	0.005
Ave of Method 1, 2 and 3	**-0.28**	**0.28**	**-0.26**	**0.26**	**-0.008**	**0.008**
Standard deviation	**0.24**		**0.19**		**0.008**	

Table 35. Soybean Proximate Tolerance Limits

Method	Protein (%)		Oil (%)	
	LCL	UCL	LCL	UCL
Method 1	-0.43	0.43	-0.36	0.36
Method 2	-0.53	0.53	-0.41	0.41
Method 3	-0.33	0.33	-0.25	0.25
Ave of Method 1, 2 and 3	**-0.43**	**0.43**	**-0.34**	**0.34**
Standard deviation	**0.33**		**0.28**	

Tables 34 and 35 show the results of tolerance limits calculation from three methods. The tolerance limits by average of three methods are used to decide whether the differences between new results (current measurements) and averages of first three measurements of single sample are still acceptable. The tolerances for standard deviation that were calculated after outlier removal are also shown in Tables 34 and 35 for corn and soybeans, respectively. An example of tolerance limits application and bias calculation is shown in the next chapter.

The proximate analysis results of repeated samples documented in the standardization spreadsheet. Notes are recorded, when biases are applied. However, they are also available in the Access database. A chart of number of measurements vs. standard deviation of repeated samples is built in the standardization spreadsheet to know the consistency of measurements done by proximate lab. A control chart of differences between every measurement and average of first three measurements with fixed tolerance limits (from table 34 and 35) is also constructed in the yearly standardization spreadsheet. Out of control data is detected by this chart.

Amino Acids

Amino Acids data are outsourced from University of Missouri. Some samples are resubmitted to the chemistry lab for quality control purposes, in the same way as for the proximates. The standard deviations and coefficient of variations are calculated for each amino acid. The 95% confidence level of tolerance limits were calculated based on long term historical data using equation 13.

Tolerance of differences from true value = $\pm\ 2 \times \sqrt{2 \times \sigma^2}$ (eq.13)

σ^2 = variance of measurements

(Mullins, 2003).

Thakur (2006) defined five key Amino Acid as the ones essential for both swine and poultry nutrition. These are threonine, cysteine, methionine, lysine and tryptophan. Tolerance limits for the five key amino acids, total sulfur (Cysteine and Methionine) and total amino acids of corn and soybeans are summarized in table 36. These tolerance limits is used to evaluate the differences between average of long term and current measurements. Three repeated samples of corn and six repeated samples of soybeans were used for amino acids quality control. The repeated samples have two to eight measurements. The complete tolerance limits for each Amino Acid can be found in Appendix G. The chart of replicates standard deviation is also built for both corn and soybeans Amino Acids.

Table 36. Corn and Soybean Amino Acids Tolerance Limits

	Corn		Soybeans	
Amino Acids	**LCL (%)**	**UCL (%)**	**LCL (%)**	**UCL (%)**
Threonine	-0.04	0.04	-0.05	0.05
Cysteine	-0.03	0.03	-0.08	0.08
Methionine	-0.04	0.04	-0.06	0.06
Lysine	-0.05	0.05	-0.10	0.10
Tryptophan	-0.03	0.03	-0.19	0.19
Total Sulfur AA	-0.06	0.06	-0.12	0.12
5 Key AA	-0.13	0.13	-0.31	0.31
Total AA	-0.82	0.82	-1.32	1.32

The hard copies of amino acid results from the external laboratory are documented in the folders by year. The amino acids data are then recorded in the spreadsheet, converted into

15% and 13% moisture basis for corn and soybeans, respectively. The converted data then are moved and saved in the “Amino Acid” table of corn or soybeans Microsoft Access databases. The quality control data and the chart are stored in the spreadsheet. However, these data are also available in the Access databases.

Fatty Acids

Fatty Acids data are outsourced from the Agronomy Department, Iowa State University. As with other outsourced tests, samples are resubmitted to the chemistry lab for quality control purposes. This data are recorded in the spreadsheet. The standard deviations and coefficient of variations are calculated for each type of fatty acids. The tolerance limits were calculated based on long term historical data using equation 13.

Table 37 contains the tolerance limits for soybean fatty acids. These tolerance limits is used to evaluate the differences between average of long term and current measurements. Eighteen samples were used as repeated samples for quality control purposes. Each sample had two measurements. The chart of replicates standard deviation is built to document reproducibility.

Table 37. Soybeans Fatty Acids Tolerance Limits

Fatty Acid	LCL (%)	UCL (%)
Saturates	-0.80	0.80
Palmitic	-0.60	0.60
Stearic	-0.54	0.54
Oleic	-3.88	3.88
Linoleic	-3.03	3.03
Linolenic	-1.26	1.26

The Fatty Acids results are moved into "Fatty Acids" table in the soybeans chemistry Microsoft Access database. The quality control data are stored in the spreadsheet. However, quality control samples are also available in the Access databases.

The documentation process of every reference chemistry item is described in the figure 35. The summary of reference data quality control program is in tables 38 and 39.

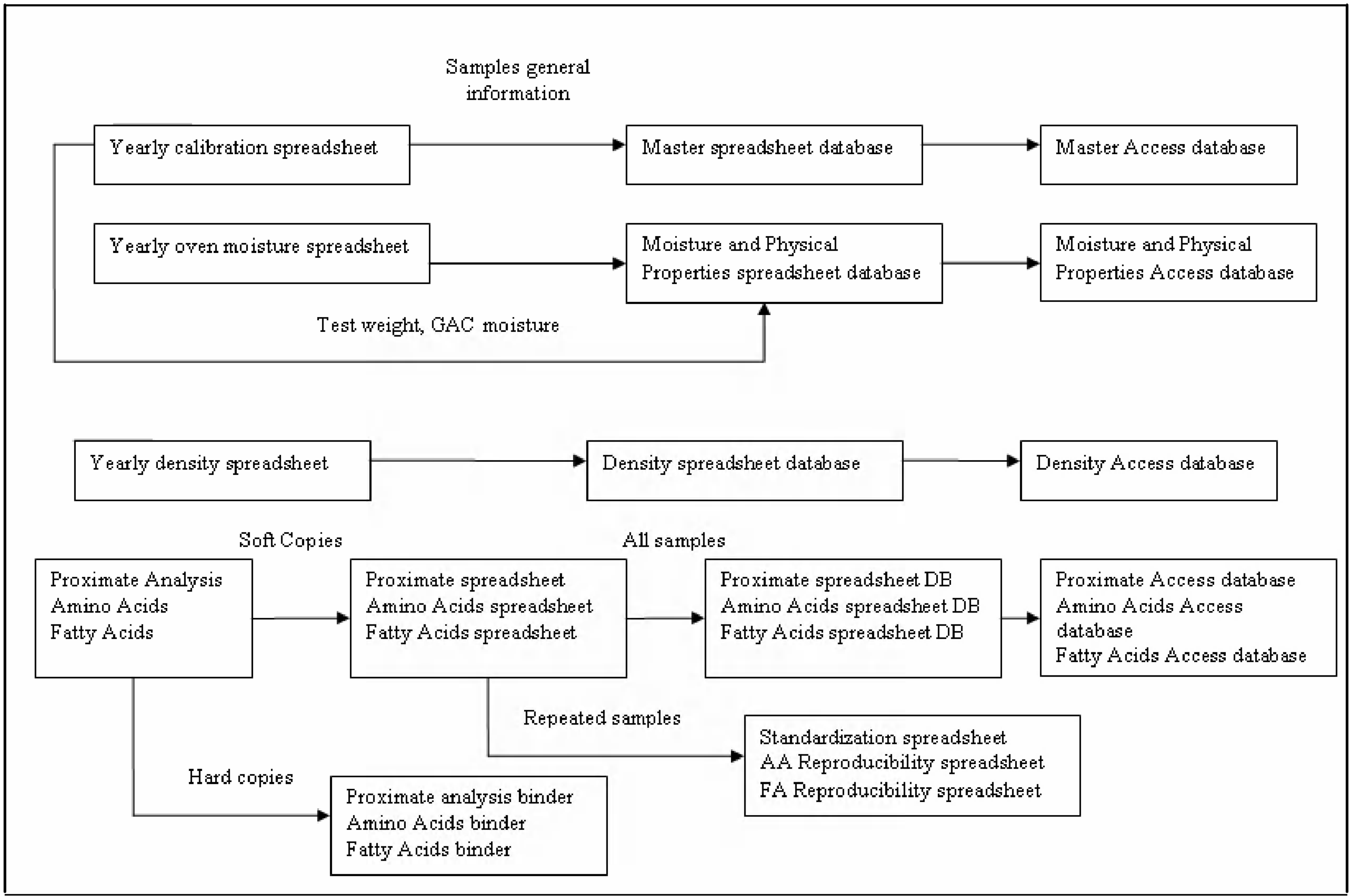


Figure 35. Reference Chemistry Documentation Process

Table 38. Internal Reference Chemistry Quality Control Program

Item	Check	Quality Control	Tolerance	Documentation	Comments
Oven Moisture	Repeatability (Precision)	Chart of ave moisture vs. ave diff. of three replicates for dishes and duplicates Chart of consecutive samples vs. std. dev (dishes and duplicates) 95% CL Control Chart of differences between dishes and duplicates.	Average difference between each pair of replicates: Corn: ≤ 0.25%, for ≤15% moisture ≤ 0.30%, for >15% moisture Soybeans: ≤ 0.20%, for all moisture Allow 5% out of control data	Spreadsheet by year "Moisture and Physical Properties" Access DB	Three replicates 10% duplication Every year, calibration samples
Corn Density	Repeatability (Precision)	Chart of std. dev of measurements: moisture = 6 replicates (GAC 2000 and 2100) test weight = 6 replicates (GAC 2000 and 2100) density = 3 replicates		Spreadsheet "Density 15%" Access DB	calibration samples
		95% CL Schewhart control chart of pyconometer daily check	Allow 5% out of control data	Spreadsheet	One chosen sample Daily check during the running of calibration samples
		Repeated samples check.	Difference between current measurements and fixed ave of first three measurments: Density = ± 0.008 g/cm^3 std. dev = 0.008 g/cm^3 pts	Spreadsheet	
		Control chart of differences of each rep and ave of first three reps Chart of number of reps vs. std. dev of every repeated samples		Spreadsheet (standardization file)	Yearly Standardization samples (30 samples)

Table 39. External Reference Chemistry Quality Control Program

Item	Check	Quality Control	Tolerance	Documentation	Comments
Corn Proximate (Protein, oil)	Repeatability Reproducibility	Repeated samples check Control chart of differences of each rep and ave of first three reps Chart of number of reps vs. std. dev of every repeated samples	Difference between current measurements and fixed ave of first three measurements: Protein = ±0.28 % std. dev = 0.24%pts Oil = ±0.26% std. dev = 0.19%pts	Hard copy (Binder) Spreadsheet "Proximate" (Access DB) Spreadsheet (standardization file)	Send at least 5% blind duplicate and 5% repeated samples when sending calibration set samples (150 samples) Yearly standardization sample (30 samples)
Soybeans Proximate (Protein, oil)	Repeatability Reproducibility	Repeated samples check Control chart of differences of each rep and ave of first three reps Chart of number of reps vs. std. dev of every repeated samples	Difference between current measurements and fixed ave of first three measurements: Protein = ±0.43 % std. dev = 0.33%pts Oil = ±0.34% std.dev = 0.28%pts	Hard copy (Binder) Spreadsheet "Proximate" (Access DB) Spreadsheet (standardization file)	Send at least 5% blind duplicate and 5% repeated samples when sending calibration set samples (150 samples) Yearly standardization sample (20 samples)
Amino acids (corn and soybeans)	Repeatability Reproducibility	Repeated samples check	see AA Table	Hard copy (Binder) Spreadsheet "AA" Access DB Spreadsheet	several chosen samples (summary of number of samples, number of reps, ave std ave and CV)
Fatty Acids soybeans	Repeatability Reproducibility	Repeated samples check	see FA Table	Hard copy (Binder) Spreadsheet "FA" Access DB Spreadsheet	several chosen samples (summary of number of samples, number of reps, ave std ave and CV)

CHAPTER 7. IMPLEMENTATION OF REFERENCE DATA QUALITY CONTROL PROGRAM

This chapter demonstrates the implementation of the reference data quality program. Oven moisture, corn density and proximate analysis quality control application are shown below:

Oven Moisture

Table 40 summarizes the quality control of corn oven moistures for 2004 and 2005. The out of control data decreased from 13.98% in 2004 to 7.38% in 2005. This may be caused by a better accuracy of balance used for oven moisture. Figure 18 in Chapter 5 shows the quality control done for balances and demonstrated the problem with the balance used for oven moistures. During months of July to August 2005, Mettler PB-153-S which is used to weigh samples for oven moisture had an accuracy problem. Resolution of this problem demonstrates the use of quality control data.

Table 40. Comparison of 2004 and 2005 Corn Oven Moisture Quality Control

	2005		2004	
	Dishes	Duplicates	Dishes	Duplicates
Ave std. dev of replicates (%pts)	0.08	0.12	0.13	0.13
Ave of CV	0.41%	0.48%	0.46%	0.49%
Out of control data	7.38%	10.00%	13.98%	9.68%
Number of samples	244	20	372	31
Average of differences (%)	0.02		-0.04	
Std. dev of differences (%)	0.17		0.31	
Out of control data (Diff)	5.26%		6.45%	

Figure 36 illustrates the precision of corn oven moisture in 2005 for dishes and duplicates. There are 3 out of control data at ≤15% moisture for the dishes. For the duplicates, there is no out of control data at ≤15% moisture basis. Figure 37 shows the repeatability of dishes and duplicates from each sample. Figure 38 is the control chart of differences between dishes and duplicates of corn oven moisture. There is only one out of control data which corresponds to 5.26%. The corn oven moisture charts can be seen in Appendix H.

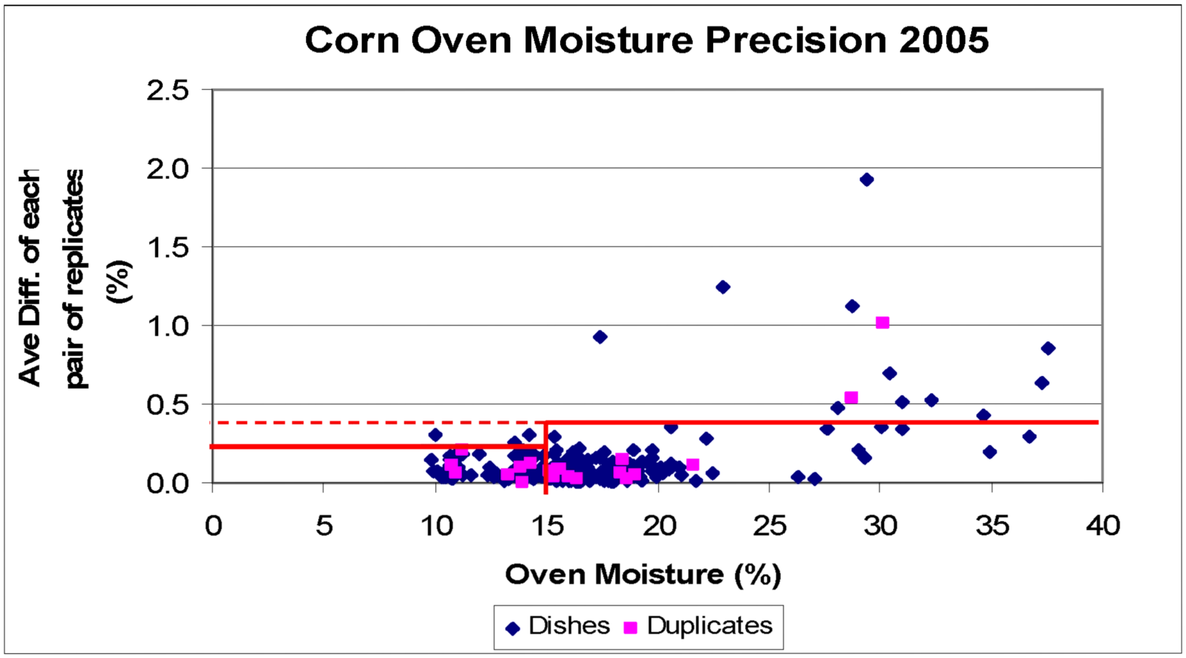


Figure 36. Corn Oven Moisture Precision Chart, 2005

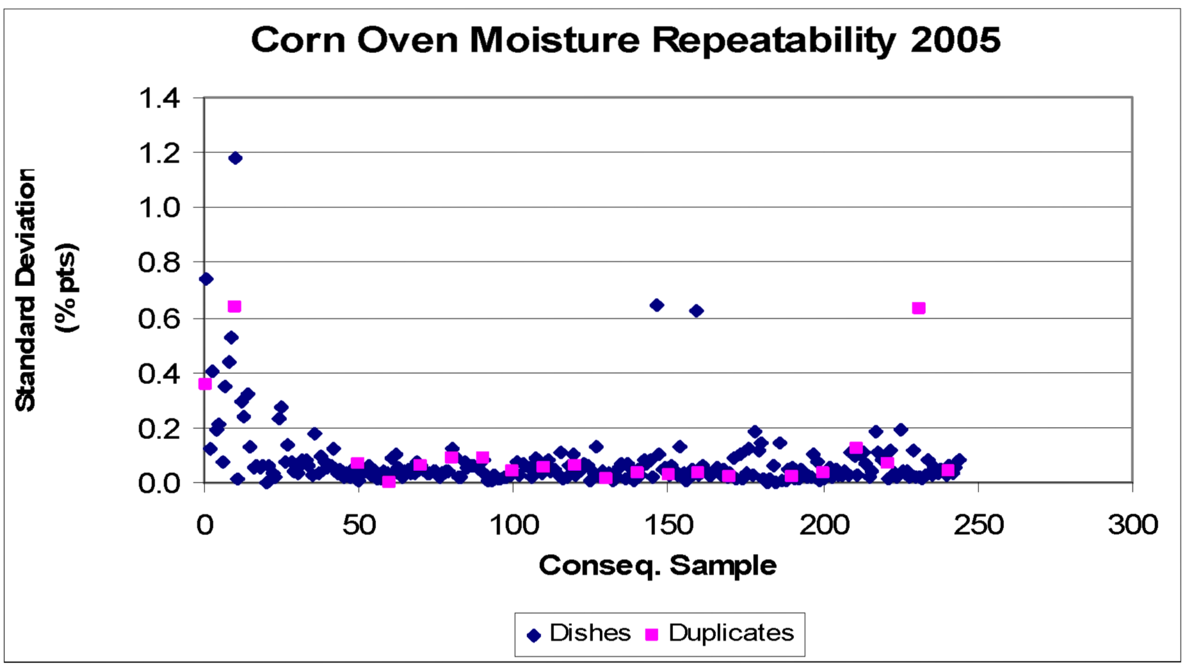


Figure 37. Corn Oven Moisture Repeatability Chart, 2005

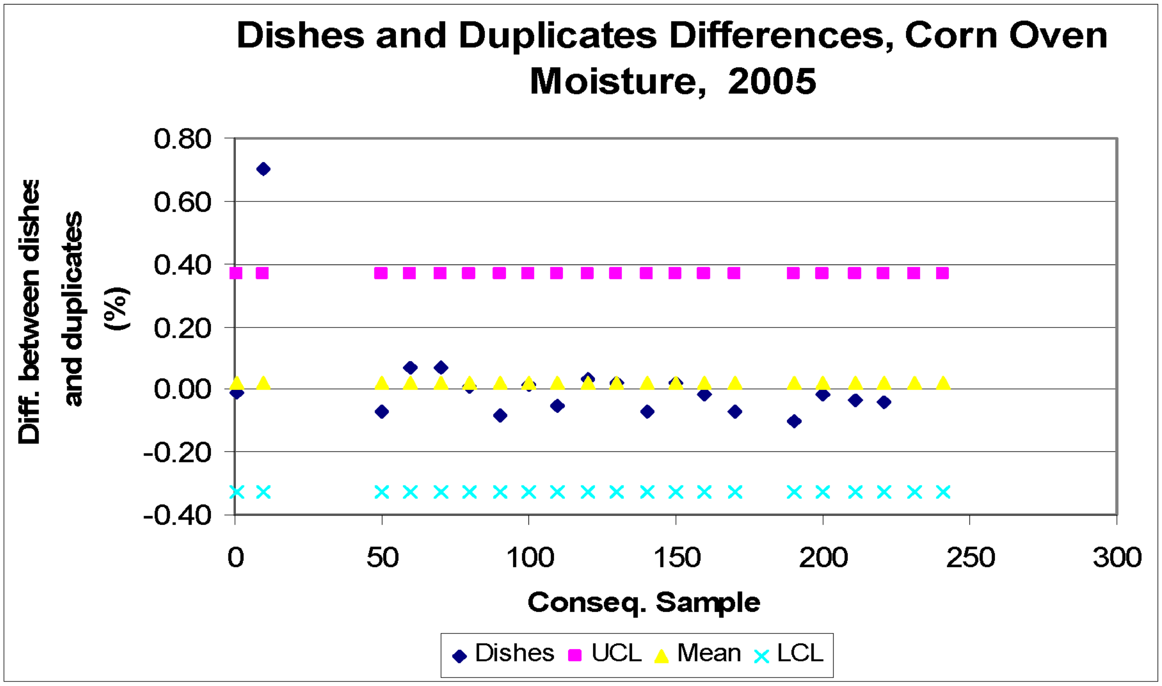


Figure 38. Differences between Dishes and Duplicate of Corn Oven Moisture Chart, 2005

Table 41 is the summary of soybeans oven moisture quality control for 2004 and 2005. Soybeans oven moisture in 2005 has a better result than in 2004. However, the out of control data is still more than 5%. This indicates that operator training and equipment check may be needed. Figure 39 shows the precision of soybeans oven moisture for dishes and duplicates. The standard deviations from three replicates of dishes and duplicates of each sample are shown in Figure 40. Figure 41 is the control chart of differences between dishes and duplicates of soybeans oven moisture. There are 2 out of control data which corresponds to 3.13%.

Table 41. Comparison of 2004 and 2005 Soybeans Oven Moisture Quality Control

	2005		2004	
	Dishes	Duplicates	Dishes	Duplicates
Ave std. dev of replicates (%pts)	0.07	0.31	0.16	0.53
Ave of CV	0.70%	3.07%	2.11%	6.17%
Out of control data	11.58%	28.33%	31.43%	40.00%
Number of samples	764	69	892	78
Average of differences (%)	-0.29		-0.08	
Std. dev of differences (%)	1.74		0.59	
Out of control data (Diff)	3.13%		5.06%	

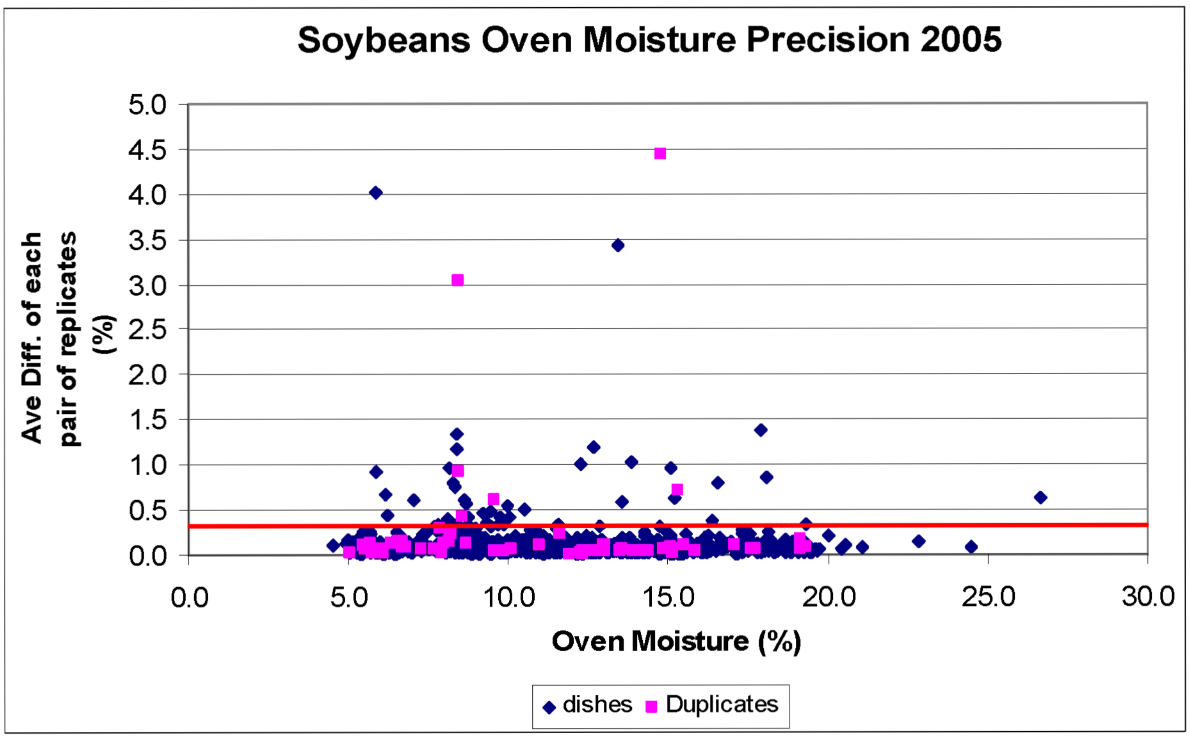


Figure 39. Soybeans Oven Moisture Precision Chart, 2005

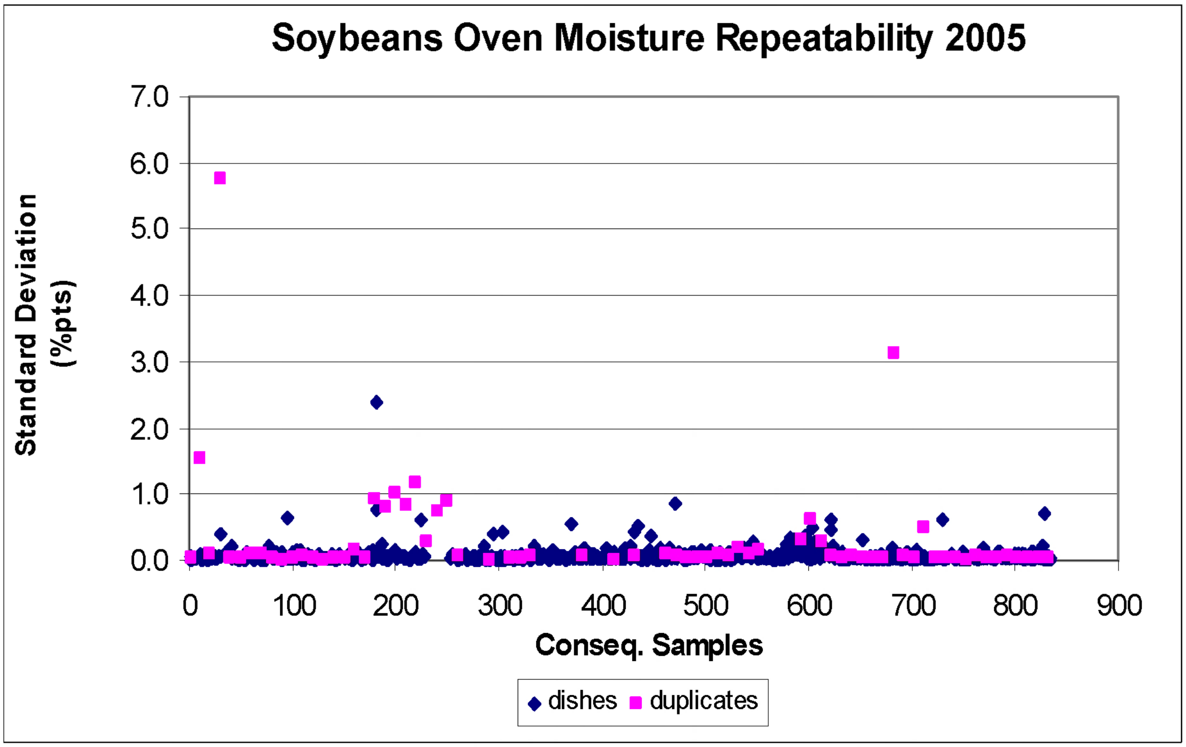


Figure 40. Soybeans Oven Moisture Repeatability Chart, 2005

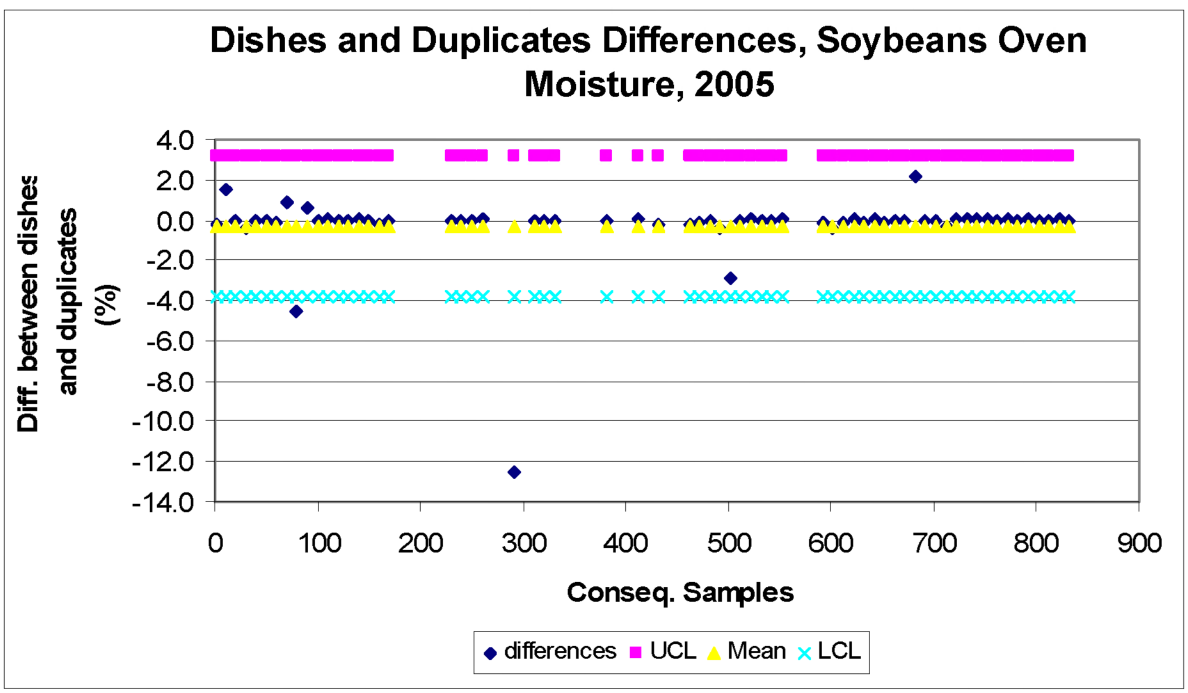


Figure 41. Differences between Dishes and Duplicate of Soybeans Oven Moisture Chart, 2005

Corn Density

Table 42. Corn Density Quality Control Summary, 2004 and 2005

	Density		Moisture		Test Weight	
	2005	2004	2005	2004	2005	2004
Std. dev of reps	0.0023	0.0018	0.16	0.19	0.40	0.57
Average CV	0.18%	0.14%	1.24%	1.72%	0.68%	0.96%
N	91	147	88	147	88	131

Table 42 is corn density quality control summary from 2004 and 2005. The two years results have nearly identical results. Figure 42 is the chart of density standard deviation from three replicates of each sample in 2005 calibration. This chart demonstrates the precision of density measurements. The pyconometer daily check using corn sample 20040049 is shown in the control chart, figure 43. There is only one out of control point. Figure 44 is the chart of number of replication versus standard deviation from standardization samples in 2005 (standardization version 11). This chart is also used to evaluate the reproducibility of measurements done by the pyconometer. The control chart of differences between each measurement and average of first three measurements of corn density from standardization samples is shown in the figure 45. The fixed tolerance limits used values from table 34. There are four out of control data from sample 19970018 and 19980018.

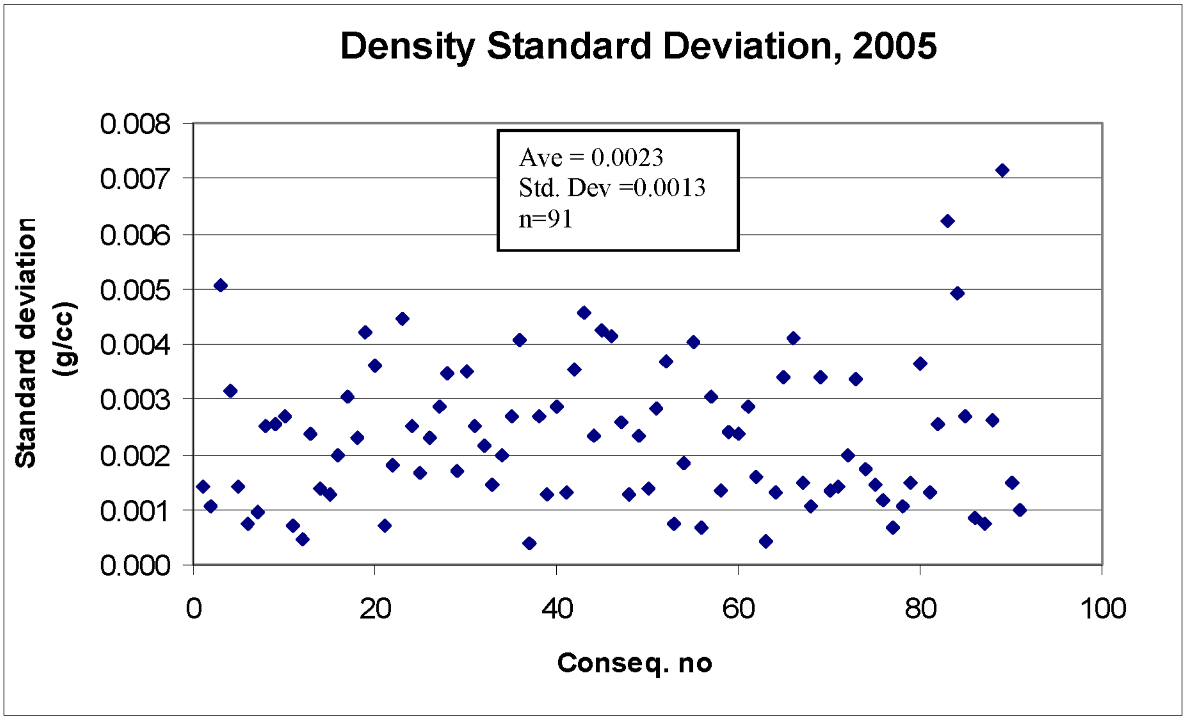


Figure 42. Density Standard Deviation Chart (from three replicates), 2005

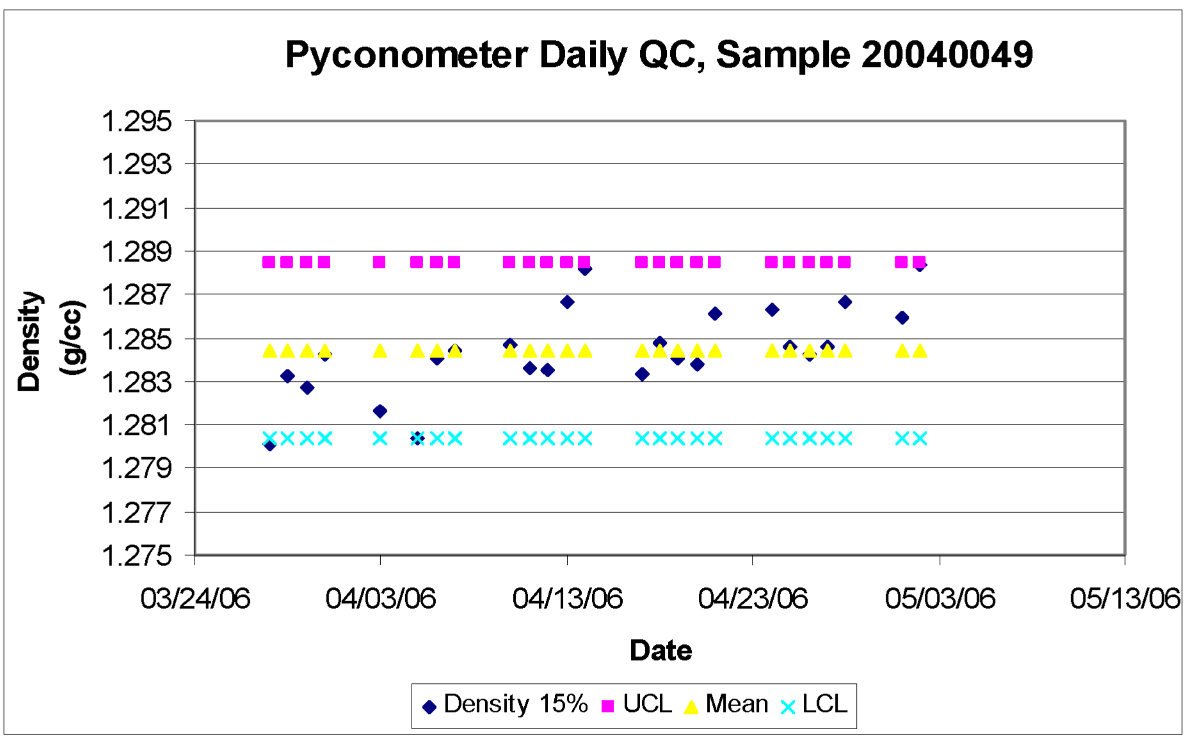


Figure 43. Pyconomenter Control Chart, Calibration 2005, Sample 20040049

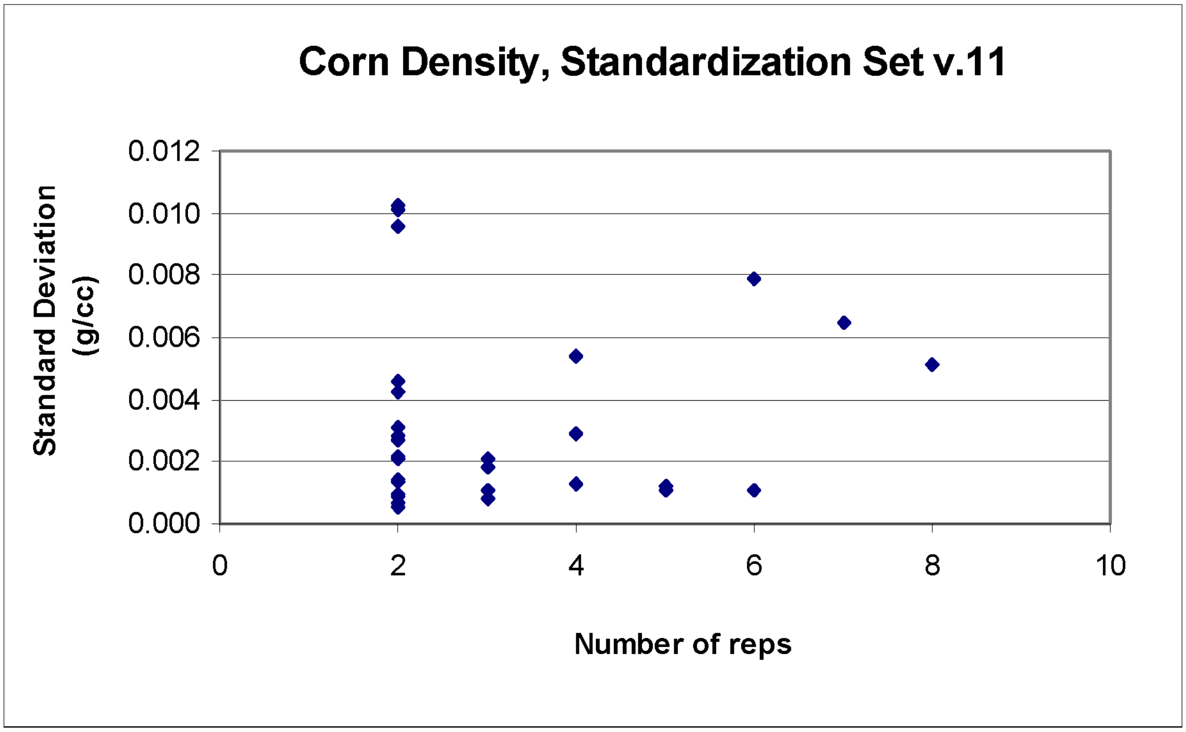


Figure 44. Number of Reps vs. Standard Deviation Chart, Corn Density Standardization v.11

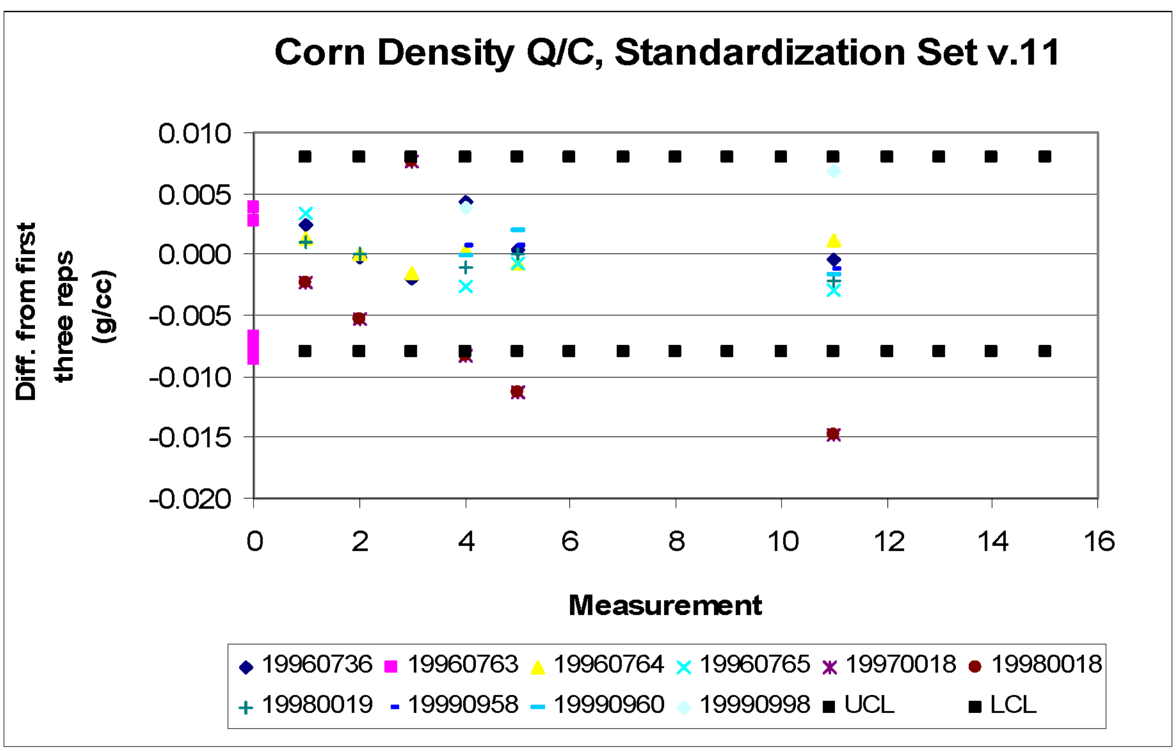


Figure 45. Corn Density Q/C, Standardization Set v.11

Proximate Analysis

Table 43. Proximate Analysis Quality Control, Standardization Set v.11

Standardization Set v.11	Corn			Soybeans	
	Protein	Oil	Density	Protein	Oil
N sample	40	40	40	31	31
Average # reps	5	5	3	7	7
Average standard deviation	0.20 %pts	0.18 %pts	0.003 g/cc	0.21 %pts	0.28 %pts
Average CV	2.49%	4.20%	0.26%	0.77%	1.17%
Average differences from average 3 reps	0.00%	-0.02%	0.000%	-0.03%	-0.03%

Table 43 summarizes the standardization sample quality control for corn and soybeans. Figures 46 and 47 show the quality control chart of corn protein from standardization samples set in 2005. Corn protein measurement is quite consistent. Standard deviation decreases when the number of measurements increases (figure 46). However, control chart which used limits from table 34 detects some out of control data (figure 47). Corn oil also indicates the same pattern (figure 48 and 49).

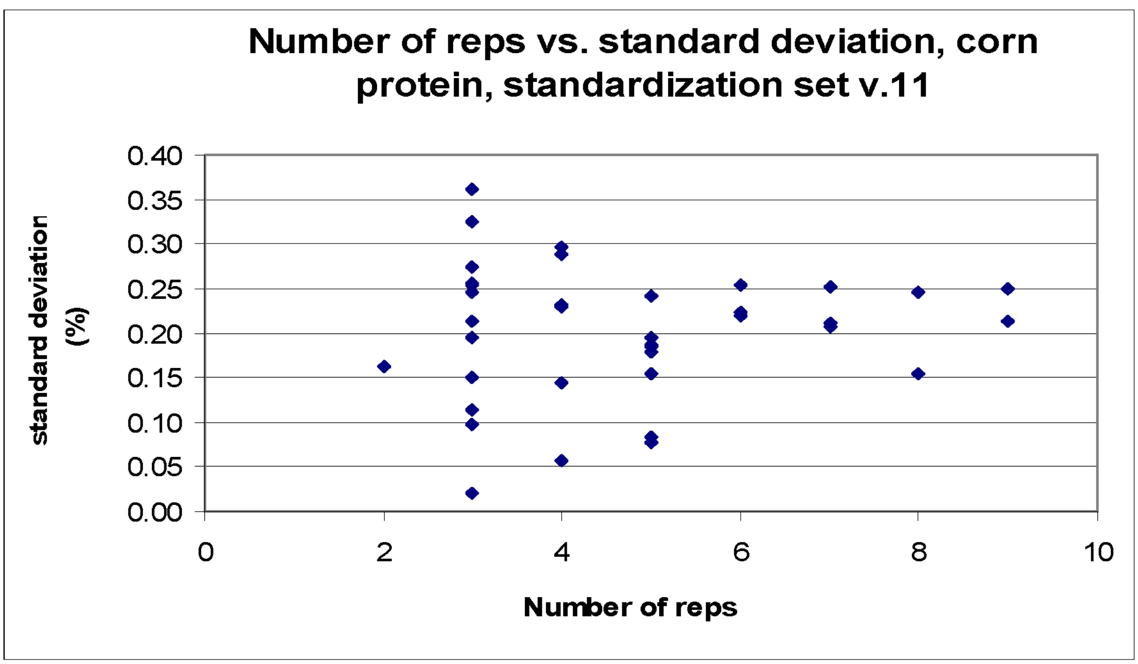


Figure 46. Number of Reps vs. Standard Deviation Chart, Corn Protein, Standardization v. 11

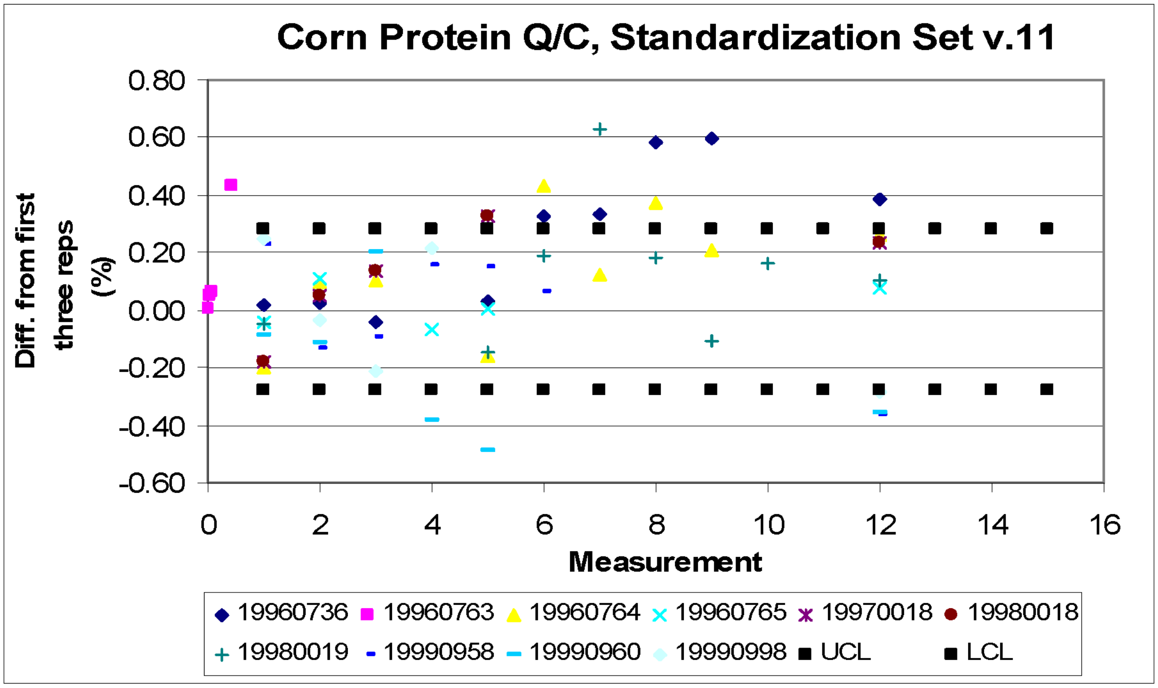


Figure 47. Corn Protein Q/C, Standardization Set v.11

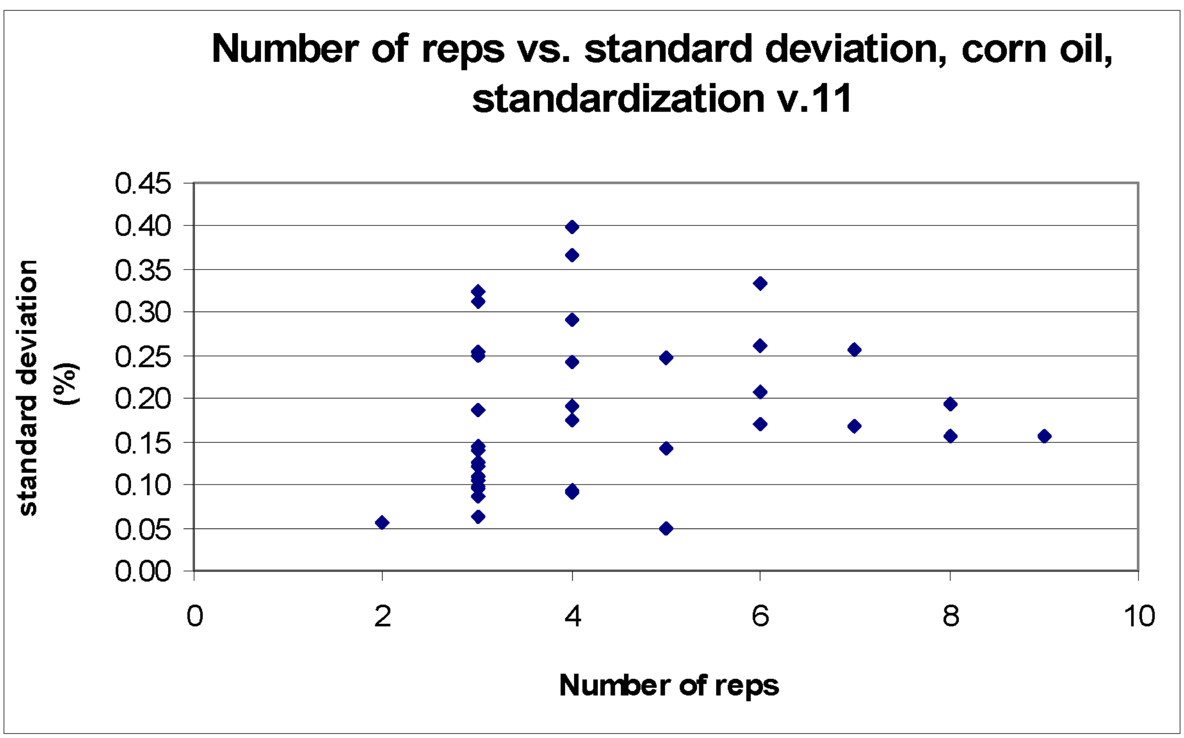


Figure 48. Number of Reps vs. Standard Deviation Chart, Corn Oil, Standardization v.11

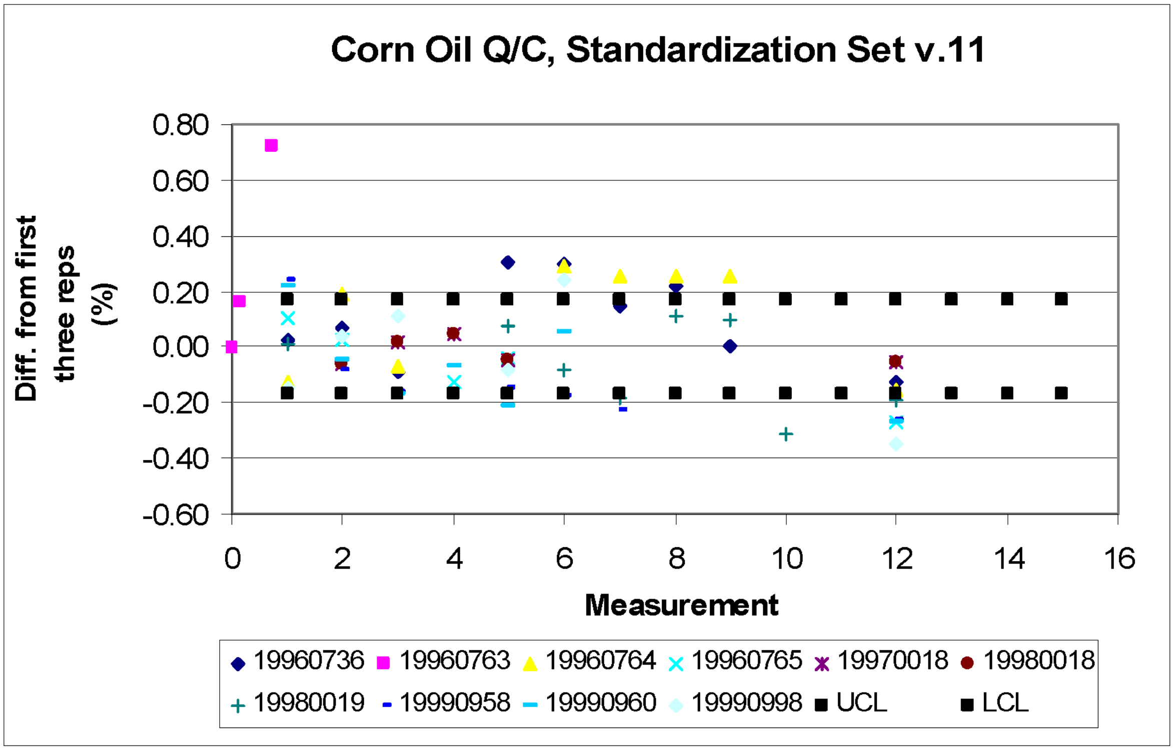


Figure 49. Corn Oil Q/C, Standardization Set v.11

Figure 50 shows the standard deviation chart of soybean protein. Some samples show a big standard deviation although the number of replications increases. The control chart in figure 51 which used tolerance limits from table 35 shows clearly that sample 19960299 has a bad reproducibility; several measurements are out of control. Soybeans oil seems to have inconsistency problem. The standard deviation also increases when number of reps increases (figure 52). Sample 19960299 also has a bad reproducibility on the oil measurements (figure 53).

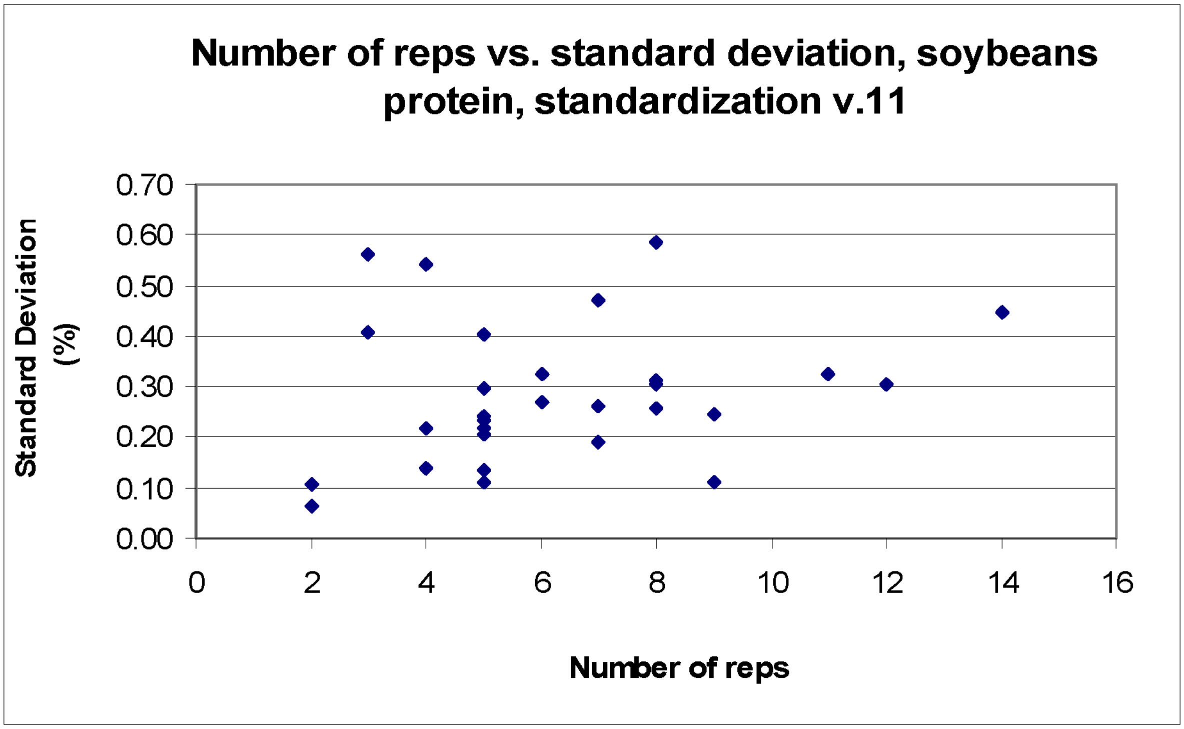


Figure 50. Number of Reps vs. Standard Deviation Chart, Soybeans Protein, Standardization v.11

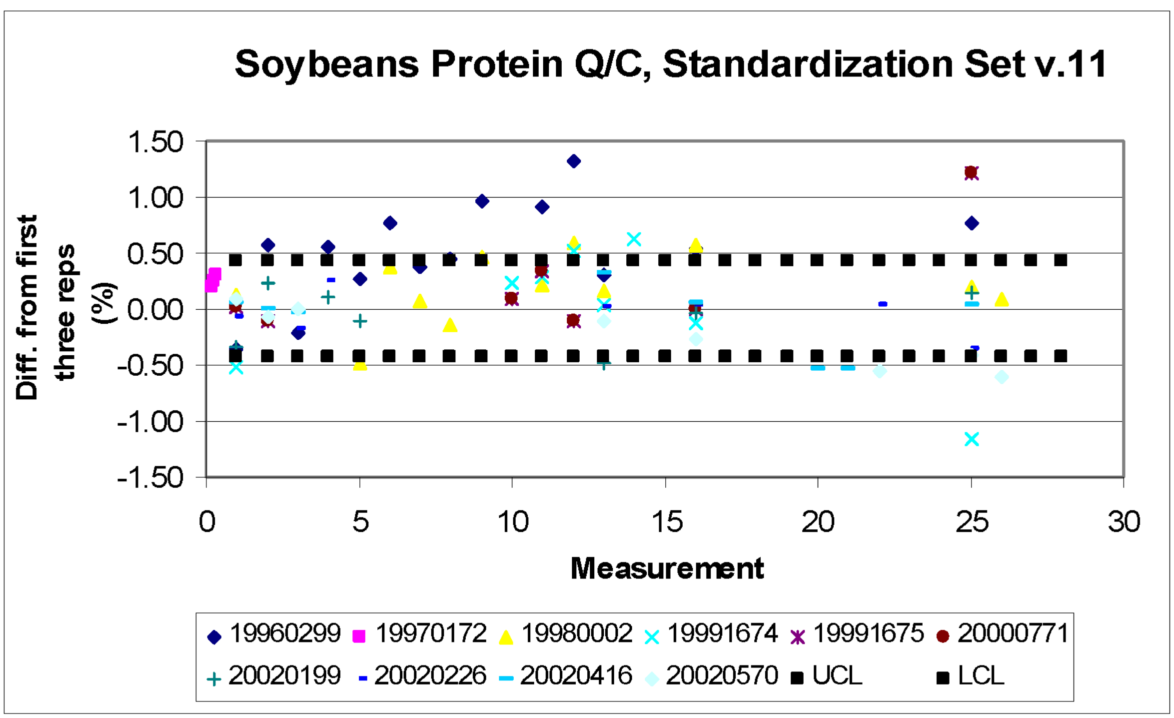


Figure 51. Soybeans Protein Q/C, Standardization Set v.11

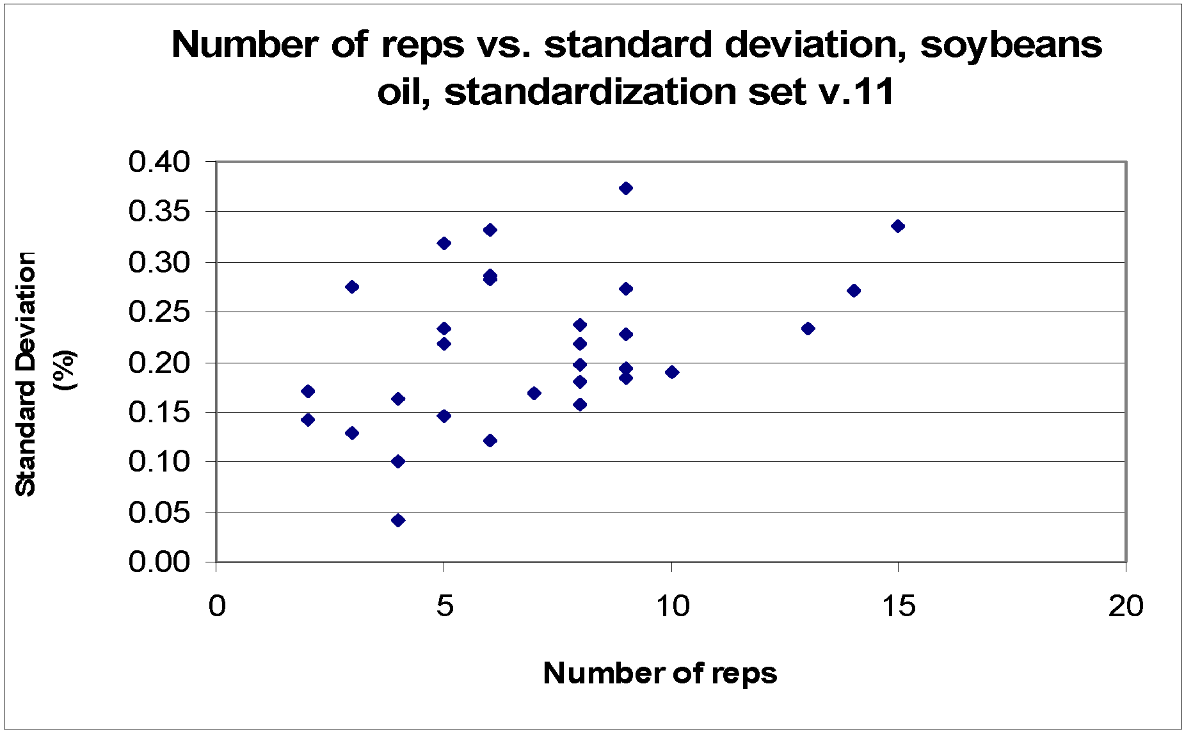


Figure 52. Number of Reps vs. Standard Deviation Chart, Soybeans Oil, Standardization v.11

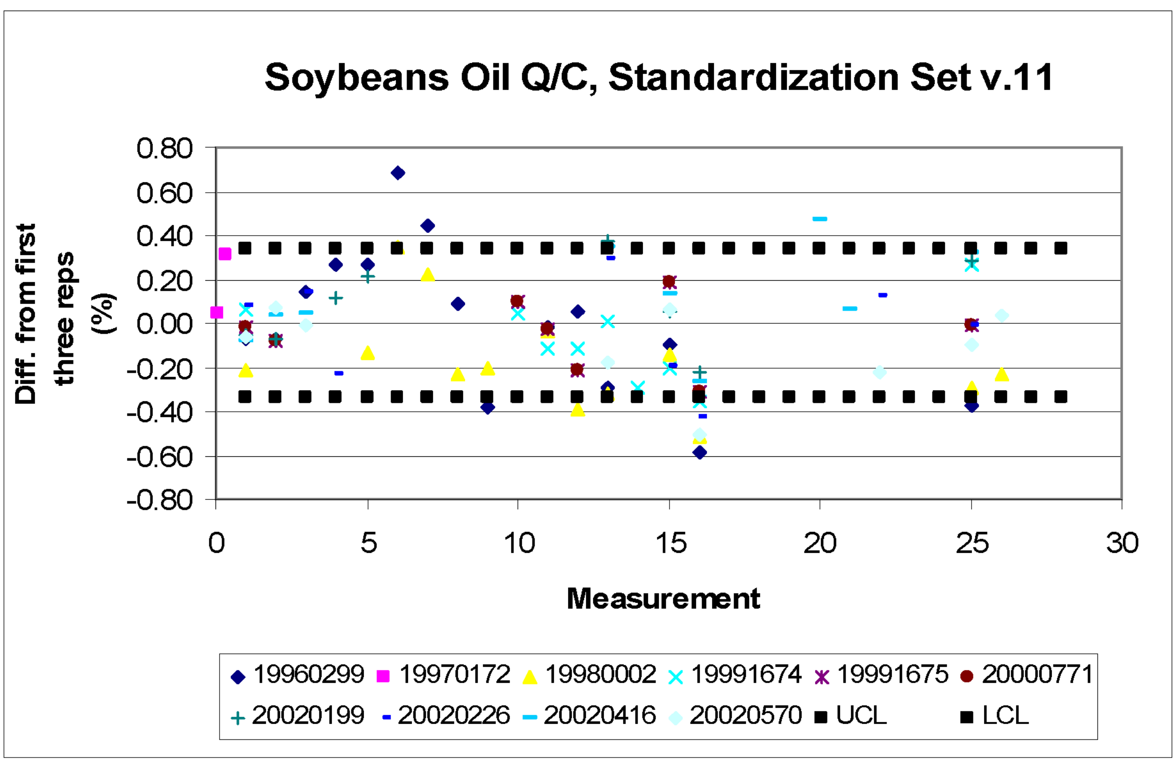


Figure 53. Soybeans Oil Q/C, Standardization Set v.11

Amino Acids

Tables 44 and 45 summarize the five key Amino Acids of corn and soybeans, respectively. Figures 54 and 55 are the five keys Amino Acids reproducibility charts of corn and soybeans. Corn has three checked samples with average of 6 reps each. Soybean has six checked samples with an average of four reps.

Table 44. Corn Amino Acids Quality Control Summary

Amino Acids	N sample	Ave reps	Ave std. dev (%)	Ave CV
Threonine	3	6	0.01	5.43%
Cysteine	3	6	0.01	6.09%
Methionine	3	6	0.02	9.06%
Lysine	3	6	0.02	7.35%
Tryptophan	3	6	0.01	22.71%
Total Sulfur AA	3	6	0.05	4.07%
TOTAL	3	6	0.02	6.24%

Table 45. Soybeans Amino Acids Quality Control Summary

Amino Acids	N sample	Ave reps	Ave std. dev (%)	Ave CV
Threonine	6	4	0.02	1.35%
Cysteine	6	4	0.03	4.60%
Methionine	6	4	0.02	4.21%
Lysine	6	4	0.04	1.60%
Tryptophan	6	4	0.07	16.14%
Total Sulfur AA	6	4	0.05	4.07%
TOTAL	6	4	0.11	2.15%

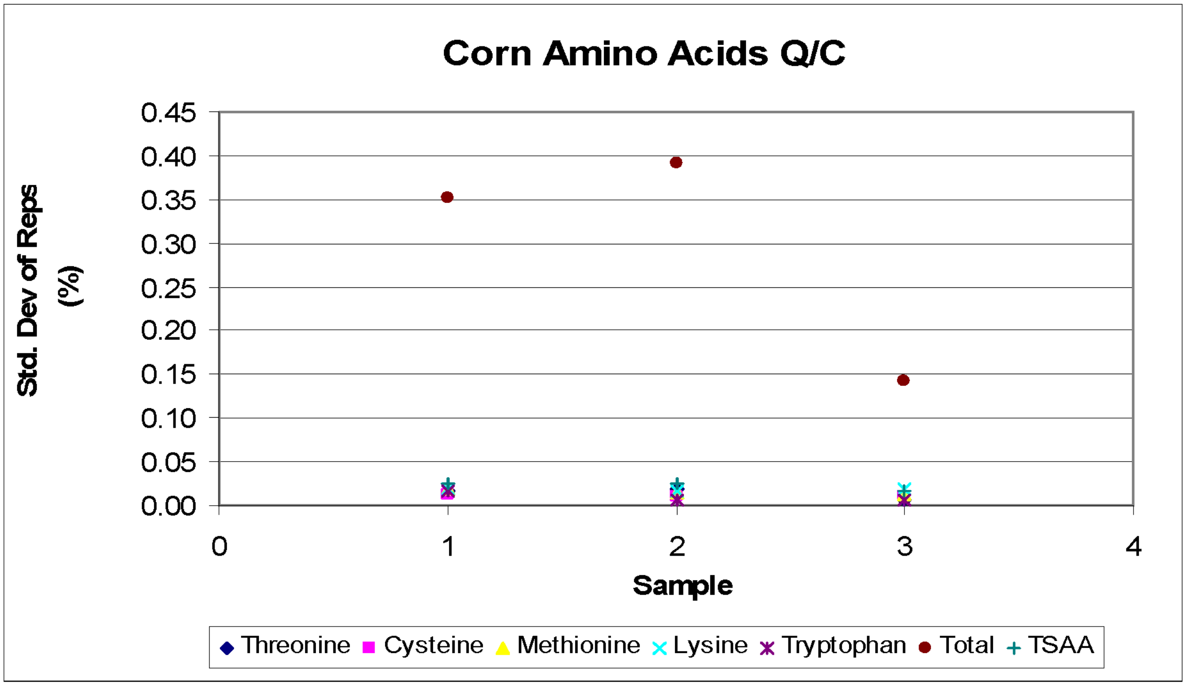


Figure 54. Corn Amino Acids Quality Control

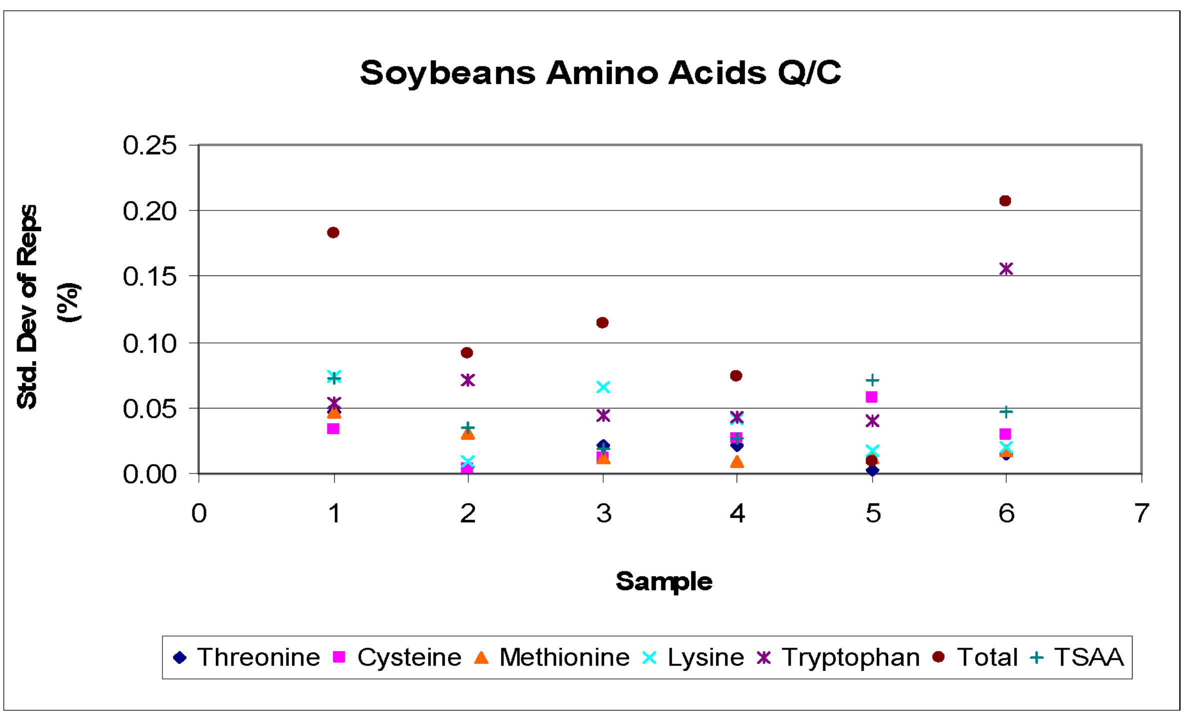


Figure 55. Soybeans Amino Acids Quality Control

Fatty Acids

Soybeans Fatty Acids quality control summary is shown in table 47. Soybeans has 44 checked samples with average of reps is two for fatty acids quality control purposes. Figure 56 is the reproducibility chart of soybeans fatty acids.

Table 46. Soybeans Fatty Acids Quality Control Summary

Fatty Acids	N	Ave reps	Ave std. dev (%)	Ave CV
Palmitic	44	2	0.34	4.78%
Stearic	44	2	0.36	6.59%
Oleic	44	2	1.36	5.09%
Linoleic	44	2	1.27	2.38%
Linolenic	44	2	0.51	9.00%
Saturates	44	2	0.60	4.73%

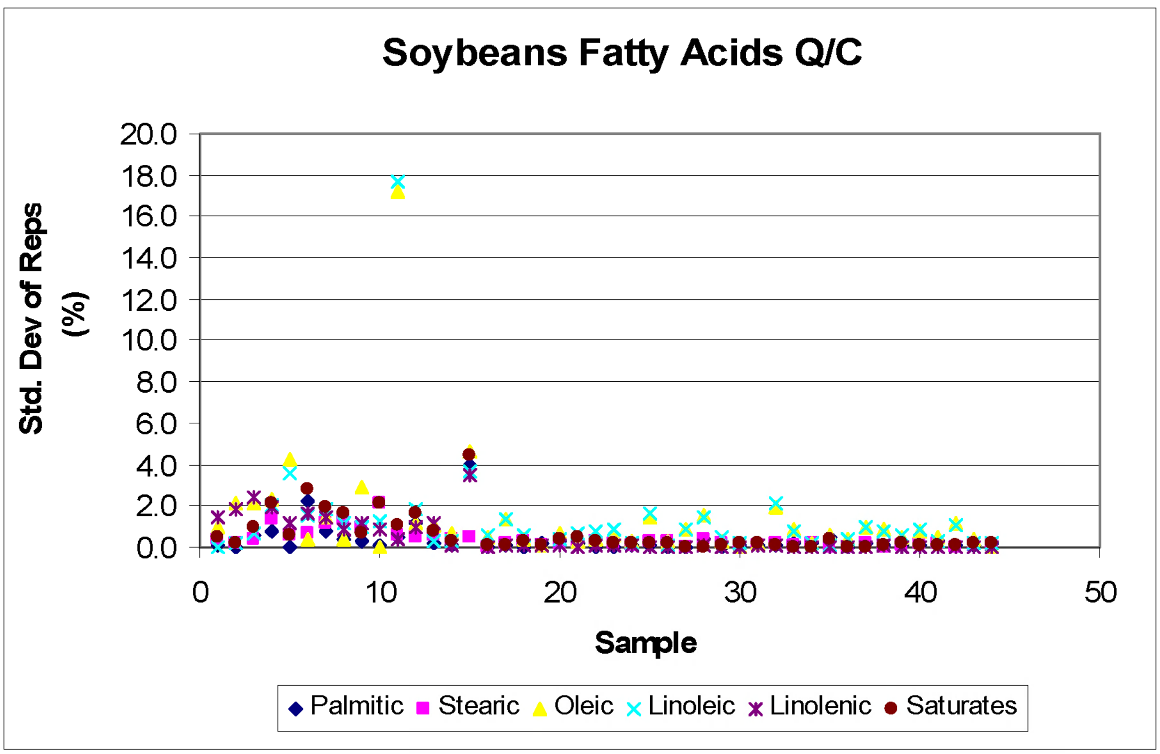


Figure 56. Soybeans Fatty Acids Quality Control

Examples of chemistry data summary from Access databases.

The examples of chemistry values quality control summary of corn and soybeans can be seen in the tables 47 and 48. These data were extracted directly from Access database which includes samples that at least have two measurements from 1993 to 2005. The average of time across reps and correlation (R) were done in the spreadsheet with extracted data from Access databases.

Table 47. Corn Chemistry Quality Control Summary

Factor	Ave (%)	Range (%)	Ave std. dev (%)	Ave CV	n samples	Ave n reps/ samples	Ave time across reps	Ave R values and time
Protein	8.41	5.00-16.48	0.19	2.23%	229	3	366*	-0.14
Oil	4.54	1.96-11.00	0.18	4.13%	229	3	366*	-0.21
Starch	59.37	48.79-63.98	0.88	1.49%	46	2	476*	0.39
Density	1.274	1.191-1.342	0.010	0.75%	165	3	no date	
Amino Acids								
Cysteine	0.18	0.16-0.21	0.01	6.37%	3	6	157*	-0.25
Lysine	0.24	0.20-0.27	0.02	7.85%	3	6	157 *	-0.02
Methionine	0.17	0.14-0.22	0.02	9.46%	3	6	157 *	-0.38
Threonine	0.25	0.23-0.28	0.02	5.93%	3	6	157 *	0.47
Tryptophan	0.05	0.01-0.06	0.01	21.90%	3	6	157 *	-0.23

*days

Table 48. Soybeans Chemistry Quality Control Summary

Factor	Ave (%)	Range (%)	Ave. std. dev (%)	Ave CV	n samples	Ave n reps/ samples	Ave time across reps	Ave R values and time
Protein	36.54	29.07-47.51	0.41	1.13%	519	3	241*	0.06
Oil	18.29	12.90-24.95	0.29	1.61%	519	3	241*	0.04
Fiber	4.32	2.70- 6.24	0.27	6.41%	183	2	271*	-0.47
Amino Acids								
Cysteine	0.59	0.45-0.79	0.04	6.13%	52	2	300*	-0.42
Lysine	2.19	1.87-2.56	0.09	4.05%	52	2	300*	0.05
Methionine	0.50	0.42-0.60	0.02	4.86%	52	2	300*	-0.25
Threonine	1.35	1.16-1.56	0.04	2.94%	52	2	300*	-0.01
Tryptophan	0.44	0.28-0.61	0.03	7.51%	52	2	300*	0.62
Fatty Acids								
Palmitic	10.59	2.80-27.70	0.34	4.78%	44	2	no date	
Stearic	5.78	2.30-27.10	0.36	6.59%	44	2	no date	
Oleic	25.78	12.90-44.30	1.36	5.09%	44	2	no date	
Linoleic	53.76	36.50-69.40	1.27	2.38%	44	2	no date	
Linolenic	4.06	0.89-12.70	0.51	9.00%	44	2	no date	
Saturates	16.37	5.30-35.70	0.60	4.73%	44	2	no date	

*days

Example of tolerance limits application and bias calculation on proximate analysis.

ISU-GQL sent 94 soybeans samples of 2004 calibration samples to the chemistry lab in May 26, 2005. There were five repeated samples (about 5% of total samples) that were included in the submission. Table 49 contains the soybean protein long term history from five repeated samples. Sample 20030683 cannot be included in the bias calculation because the differences between each replicates are not within tolerance limits. This sample only has three measurements. Table 50 shows the results of soybean protein bias calculation. Two samples are within tolerance and the other two are not within tolerance. The average of differences was -0.38, this value was within tolerance limits (±0.43). However, the standard deviation was 0.39; this value is not within a tolerance (0.33). Because the variability (standard deviation) is quite high, the decision to apply bias was not made**.**

Table 49. Soybeans Protein Long Term History

Sample No	P1 (%)	P2 (%)	P3 (%)	Ave (%)	Std. Dev (%)	P1-P2 (%)	P1-P3 (%)	P2-P3 (%)
20020226	37.04	36.99	36.95	36.99	0.05	0.05	0.09	0.04
20020417	36.88	36.92	36.92	36.91	0.02	-0.04	-0.04	0.00
20030026	37.89	37.76	38.01	37.89	0.13	0.13	-0.12	-0.25
20030028	36.37	36.25	36.62	36.41	0.19	0.12	-0.25	-0.37
20030683	37.58	38.60	38.53	38.09	0.57	-1.02	-0.95	0.07

Table 50. Soybeans Protein Bias Calculation

Sample No	Average of Three (%)	Current Protein (%)	Differences (%)
20020226	36.99	36.45	-0.54
20020417	36.91	36.07	-0.84
20030026	37.89	37.72	-0.16
20030028	36.41	36.46	0.04
20030683		38.39	
Average			**-0.38**
Std. dev			**0.39**

Table 51 is the soybeans oil long term history from five repeated samples. Table 52 shows the results of bias calculation. Two samples are within tolerance and three samples are not within tolerance. The average of differences is 0.36; this value is not within tolerance limits (±0.34). The standard deviation is 0.19; this value is within tolerance (0.28). The decision to apply a bias of -0.36 to soybeans oil of all submitted samples was also made. The smaller the standard deviation, the stronger the decision.

Table 51. Soybeans Oil Long Term History

Sample No	O1 (%)	O2 (%)	O3 (%)	Ave (%)	Std. Dev (%)	P1-P2 (%)	P1-P3 (%)	P2-P3 (%)
20020226	18.41	18.53	18.54	18.49	0.07	-0.12	-0.13	-0.01
20020417	18.69	18.85	19.07	18.87	0.19	-0.16	-0.38	-0.22
20030026	17.21	17.27	17.00	17.16	0.14	-0.06	0.21	0.27
20030028	18.41	18.53	18.67	18.54	0.13	-0.12	-0.26	-0.14
20030683	18.85	18.45	18.53	18.65	0.21	0.40	0.32	-0.08

Table 52. Soybeans Oil Bias Calculation

Sample No	Average of Three (%)	Current Oil (%)	Differences (%)
20020226	18.49	18.96	0.47
20020417	18.87	19.03	0.16
20030026	17.16	17.58	0.42
20030028	18.54	19.15	0.62
20030683	18.65	18.80	0.15
Average			0.36
Std. dev			0.19

CHAPTER 8. COST OF QUALITY IN ISU-GQL

This section identifies and estimates costs associated with quality. By evaluating cost of quality in the lab, ineffective activities can be reduced and service quality will improve. In the end, ISU-GQL can save money.

According to Gryna (1999), the costs of quality can be categorized into four items:

1. *Internal failure costs* are the costs of deficiencies discovered before delivery which are associated with the failures (nonconformities) to meet explicit requirements or implicit needs of external and internal customers.
2. *External failure costs* are the costs associated with deficiencies that are found after product is received by the customer.
3. *Appraisal costs* are the costs incurred to determine the degree of conformance to quality requirement.
4. *Prevention costs* are the costs incurred to keep failure and appraisal costs to minimum.

The costs of quality in the ISU-GQL were based on the yearly calibration of NIRS instruments and service to the customer activities. The costs of quality in ISU-GQL were divided into three of the four categories: 1) prevention and 2) failure (external and internal) and 3) appraisal costs. In some cases, there was insufficient numerical data to make complete cost estimates. For these, some general assumptions were made:

- The samples used for calibration are 300 samples (150 corn and 150 soybeans).
- ISU- GQL analyzes 8000 corn samples/year and 14000 soybeans samples/year. Ten percent of the samples in all tests are duplicated for quality control purposes.
- ISU-GQL sends 30 samples/submission to the chemistry lab.

- On average, four blind duplicate samples per submission are sent to the chemistry lab.
- On average, four repeated samples per submission are sent to the chemistry lab.
- Student labor is able to scan 50 samples/hour using an NIRS instrument.
- ISU-GQL does oven moisture on 500 corn samples and 500 soybean samples every year. Ten percent are duplicated for quality control purposes. An employee needs 12 minutes to do oven moisture on one sample (5 samples/hour).
- The trainers were graduate labor.
- It needs 20 hours to work on wrong sample ID every year.
- It needs 20 hours to work on missing data every year.
- The unplanned downtime equipment or instrument happens 5 times/year.
- When the variability with clear error happens on moisture measurement, test weight and seed counting, these activities are repeated to all samples (corn and soybeans). Employee needs 15 minutes/sample (4 samples/hr) to do these activities.
- When the variability with clear error happens on NIRS instruments analyses, the sample scanning is repeated for all samples (corn and soybeans) on all instruments (13).
- When the variability of measurements (clear errors) happened on reference values, all samples (corn and soybeans) are resent to the chemistry lab.
- Unplanned downtime equipment/instruments means an NIR instruments used for service cannot be used to analyze customer samples:
    - ISU-GQL has to pay labor for 8 hours because labor cannot work for the whole day.

- ISU-GQL cannot do service to customers (8 hours * 50 samples/hour = 400 samples).

- Suppliers lost and customers lost do not happen in the ISU-GQL.
- One year has 240 working days (48 weeks).

Table 53 contains values used for cost calculation. This information was received from the ISU-GQL laboratory manager.

Table 53. Values used for Cost Calculations

Items	Price
Service using NIR instrument/sample	$4.75
Undergraduate Labor/hour	$10.00
Graduate Labor/hour	$18.00
Soybean/sample –Eurofins (Protein, Oil)	$20.00
Corn/sample –Eurofins (Protein, Oil, Starch)	$30.00

(Rippke, personal communication, 2006)

The calculations of quality cost are shown in tables 54 and 55. Prevention activities cost $15,924. The daily NIRS check sample and QC data management and processing have a biggest expenses among the others activities ($9600). Sending blind duplicates and repeated samples costs $2000. Therefore, the quality control activities must be done effectively. The appraisal and failures cost $20,738. And the total cost of quality in ISU-GQL is $36,662. This will occur if the prevention activities are not performed well. However, if the prevention activities can be done successfully, the failure costs can be reduced or even eliminated. Then, the total costs of quality might be reduced by more than half.

Table 54. Prevention Costs

Operation	Prevention	Cost
1. Calibration of NIRS Instruments		
a. Prepare samples for calibration	Prepare sample ID Data handling and management Sample storage, handling and retrieval Train operators Trainer cost	$10/hr*5 hrs*1 labor = $50 $10/hr*5 hrs*1 labor = $50 $10/hr*5 hrs*1 labor= $100 $10/hr*2 hrs*2 labors = $40 $18/hr*2 hrs*1 labor = $36
b. Measure chosen samples moisture (GAC), test weight of 200 seeds	Equipment check and maintenance (QC) Train operators Trainer cost	$10/hr*2hr/wk*48 wks*1 labor = $960 $10/hr*2 hr*2 labors = $40 $18/hr*2 hr*1 labor = $36
c. Oven Moisture activity	Duplicate 10% of the samples Train operators Trainer cost	$10/hr*100samples/(5samples/hr)*1 labor= $200 $10/hr*1hr*2 labors= $20 $18/hr*1hr*1 labor= $18
d. Scan chosen samples using NIRS units (300) Scan standardization samples using NIRS units (20 soybeans, 30 corn) Scan temperature stabilization samples using NIRS units (80 samples)	Check NIRS instruments daily Make duplication of service samples (10% of the total samples/submission) QC data management and processing Train operators Trainer cost	$10/hr* 2 hrs*240 days*1 labor= $4800 $10/hr* 0.1*22000 samples/(50 samples/hr)* 1 labor= $440 $10/hr*10 hrs/wk*48 wks*1 labor= $4800 $10/hr*3 hrs*2 labors= $60 $18/hr*3 hr*1 labor= $54
e. Organize Spectral Data	Train people to do data storage and processing Trainer cost	$18/hr*10 hrs*2 labors = $360 $18/hr*10 hrs*1 labor = $180
f. Send yearly crop samples to chemistry lab (reference values)	Send blind duplicates (5%/more of total samples/submission) Send repeated (standardization) samples (5%/more of total samples/submission)	($20/samples*20samples)+ ($30/samples*20samples) = $1,000 ($20/samples*20samples)+ ($30/samples*20samples) = $1,000
g. Organize Reference values data	Train people to do data storage and processing Trainer cost	$18/hr*10 hrs* 2 labors= $360 $18/hr*10 hrs* 1 labor= $180
h.Do calibrations of NIRS instruments and analyze the result	Train people to do calibration Trainer cost	$18/hr* 10 hrs*2 labors= $360 $18/hr*10 hrs* 1 labor= $180
i. Document activities during calibration	Train people Trainer cost	$18/hr* 10 hrs*2 labors= $360 $18/hr*10 hrs* 1 labor = $180
2. Service to customers		
a. Scan customer's samples using NIRS units	Check NIRS instruments daily Make duplication of service samples (10% of the total samples/submission)	already calculated (1 d)
b. Data results organization	Train people to do data storage Trainer cost	$10/hr*2 hrs*2 labors= $40 $10/hr*2 hrs*1 labor= $20
c. Send results to customers		
TOTAL		**$15,924**

Table 55. Appraisal and Failures Costs

Operation	Appraisal and Failures	Cost
1. Calibration of NIRS Instruments		
a. Prepare samples for calibration	Wrong sample ID Missing data	$10/hour*20 hours*1 labor= $200 $10/hour*20 hours*1 labor= $200
b. Measure chosen samples moisture, test weight of 200 seeds	Variability of measurements (with clear error)	$10/hr*300samples/(4 samples/hr)*1 labor= $750
c. Oven Moisture activity	Variability of measurements (with clear error) Missing data	$10/hour*20 hours*1 labor= $200 $10/hour*20 hours*1 labor= $200
d. Scan chosen samples using NIRS units (300) Scan standardization samples using NIRS units (20 soybeans, 30 corn) Scan temperature stabilization samples using NIRS units (80 samples)	Variability of measurements (with clear error) Unplanned downtime equipment/ instrument	$10/hour* 430 samples/(50 samples/hour)* 13 instruments*1 labor= $1118 $10/hour*8 hour*5 times*1 labor= $400 $4.75/samples*400 samples* 5 times= $9500
e. Organize Spectral Data	Missing data	$10/hours*20 hours*1 labor= $200
f. Send yearly crop samples to chemistry lab (reference values)	Variability of measurements (with clear error)	($20/samples*150samples)+ ($30/samples*150 samples)= $7,500
g. Organize Reference values data	Missing data	$10/hour*20 hours*1 labor= $200
h. Do calibrations of NIRS instruments and analyze the result	Failure analysis Suppliers lost	
i. Document activities during calibration	Missing data	$10/hour*20 hours*1 labor= $200
2. Service to customers		
a. Scan customer's samples using NIRS units	Customers lost	
b. Data results organization		
c. Send results to customers		
TOTAL		**$20,738**

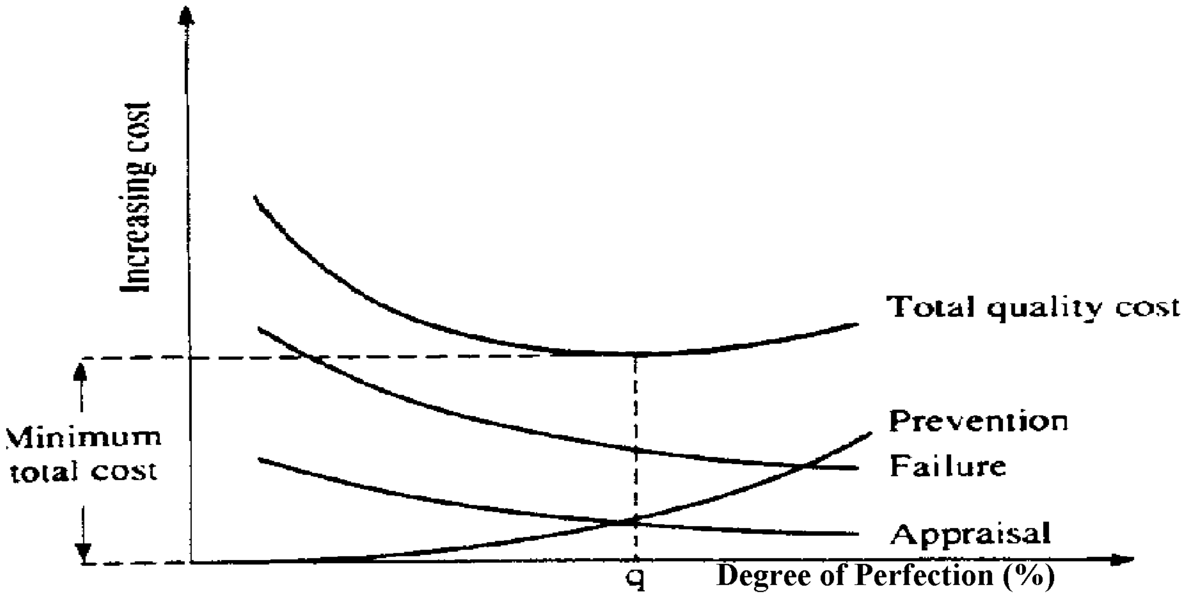


Figure 57. Cost of Quality Graph
(Source: http://www.educesoft.com/quality/costofquality.htm)

The graph on Figure 58 shows that there is a minimum total cost of quality, which is a combination of prevention, appraisal and failure. Reducing any of these reduces the total cost. The key to minimum cost, is maintaining the correct balance among the three. Clearly prevention reduces both appraisal and failure costs, however eventually the cost of prevention itself may increase the total cost. If this happen, the prevention cost must be controlled and set at an effective level.

In addition, there will be some potential benefits by doing prevention activities (quality control). When this activity is performed effectively, the lab data is possible for third party audit or accreditation. If ISU-GQL is able to get accreditation, there will be more suppliers and customers. With additional suppliers and customers, ISU-GQL will receive more revenues

CHAPTER 9. CONCLUSION AND FUTURE DIRECTION

The study of developing quality control program for NIRS instruments, supporting equipment and reference data leads to some specific conclusions as follow:

- Control charts using fixed tolerances that derived from SEP (Standard Error of Prediction) as described in AACC Method 39-00 are more appropriate than shewhart control charts because fixed tolerances come from the known performance of NIRS instruments. In addition, the quality control data itself does not influence tolerance limits as in the Schewhart control charts.
- Tolerances that come from accuracy of calibration without sample prescreening should be used for the various tests.
- Equipment quality control can improve consistency of data generated by ISU-GQL.
- Laboratory climate data provides basis to look at error pattern of other data.
- Reference data quality control only includes internal quality control. Reference data only can be verified against itself over time (reproducibility).
- ISU-GQL satisfies some requirements stated in ISO 17025. By developing a quality control program for NIRS, supporting equipment, laboratory condition and reference data, ISU-GQL meets the requirement in ISO 17025 section 5.9 about assuring the quality of test and calibration results (Quality Control). Specifically, the supporting equipment quality control program satisfies ISO 17025 section 5.5 about equipment. Quality control program for laboratory room condition meet the requirement ISO17025 section 5.3 about accommodation and environmental condition. The documentation satisfies section 4.12 of ISO 17025.
- ISU-GQL satisfies some objectives of quality control:

- ISU-GQL has upgraded the overall quality of laboratory performance with having quality control program that includes tolerance setting, procedures of quality control activities and documentation of quality control data.
- ISU-GQL is able to maintain a continuing assessment of the quality data generated by analysts.
- ISU-GQL will be able to identify good analytical methods and research needs for future developments by analyzing quality control data.
- ISU-GQL has addressed quality documentation requirements in research laboratories.
- ISU-GQL can provide a permanent record of instrument performance using documentation of instrument quality control data.
- Reference chemistry data documentation in ISU-GQL has improved.
- By developing control charts for NIRS daily check and instrument duplicates, ISU-GQL may identify training needs and possible sources of errors.

• Lab data generated by ISU-GQL is eligible for third party audit.
• The effective prevention activities will reduce the cost of quality by about 50% of total quality costs because the failures cost can be reduced or even be eliminated.

Although some objectives of quality control have been achieved, there is more work to be done. Some directions for future effort are:

• From the NIRS daily check data, NIRS instruments show a cyclic behavior and fluctuation, this pattern needs to be analyzed and investigated. Root cause analysis may be appropriate to address this problem.

- All errors including wrong quality control activities done by operators need to be recorded on weekly basis. Error recording will help to improve effective and efficient quality control activities in the future. In addition, cost of quality can be performed more accurately in a more timely manner.
- Proficiency tests need to be done for the reference data (both internal and external data) to make sure that data generated by lab are consistent with other laboratories.
- Continual revision, addition and improvement to quality control program must be done for better laboratory performance.
- A regular review of quality control data generated by the lab needs to be done. This will give opportunity for management review to analyze quality control done by lab. Better actions can be taken to perform effective and efficient activities.
- Operator training programs should include the knowledge and use of quality control data. This will give benefits in improving consistency of lab results.

APPENDIX A. NIRS TOLERANCE CALCULATION

Table 1. Infratec Corn Moisture Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200205	0.62	SECV	0.62	1.24	0.21	0.41	0.29	0.58
Base Calibration + Temp +Stabilization	CN200205	0.61	SECV	0.62	1.23	0.20	0.41	0.29	0.57
Original Calibration 2004	CN20030X	0.48	SECV	0.57	1.14	0.19	0.38	0.27	0.53
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.55	SECV	0.57	1.13	0.19	0.37	0.26	0.53
Validation 553075 (Crop 2004)	CN20030X	0.58	SEP	0.57	1.14	0.19	0.38	0.27	0.53
Validation 1241350 (Crop 2004)	CN20030X	0.45	SEP	**0.55**	**1.10**	**0.18**	**0.36**	**0.26**	**0.51**

Table 2. Infratec Corn Oil Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200205	0.42	SECV	0.42	0.84	0.14	0.28	0.20	0.39
Base Calibration + Temp +Stabilization	CN200205	0.39	SECV	0.41	0.81	0.13	0.27	0.19	0.38
Original Calibration 2004	CN20030X	0.48	SECV	0.43	0.86	0.14	0.28	0.20	0.40
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.27	SECV	0.39	0.78	0.13	0.26	0.18	0.36
Validation 553075 (Crop 2004)	CN20030X	0.37	SEP	0.39	0.77	0.13	0.25	0.18	0.36
Validation 1241350 (Crop 2004)	CN20030X	0.48	SEP	**0.40**	**0.80**	**0.13**	**0.26**	**0.19**	**0.37**

Table 3. Infratec Corn Starch Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200205	0.71	SECV	0.71	1.41	0.23	0.47	0.33	0.66
Base Calibration + Temp +Stabilization	CN200205	0.73	SECV	0.72	1.43	0.24	0.47	0.34	0.67
Original Calibration 2004	CN20030X	0.95	SECV	0.80	1.59	0.26	0.52	0.37	0.74
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.65	SECV	0.76	1.52	0.25	0.50	0.35	0.71
Validation 553075 (Crop 2004)	CN20030X	0.77	SEP	0.76	1.52	0.25	0.50	0.36	0.71
Validation 1241350 (Crop 2004)	CN20030X	0.88	SEP	**0.78**	**1.56**	**0.26**	**0.52**	**0.36**	**0.73**

Table 4. Infratec Corn Density Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200205		SECV		0.000				0.000
Base Calibration + Temp +Stabilization	CN200205		SECV		0.000				0.000
Original Calibration 2004	CN20030X	0.021	SECV	0.021	0.041	0.007	0.014	0.010	0.019
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.019	SECV	0.020	0.039	0.006	0.013	0.009	0.018
Validation 553075 (Crop 2004)	CN20030X	0.022	SEP	0.020	0.041	0.007	0.013	0.010	0.019
Validation 1241350 (Crop 2004)	CN20030X	0.024	SEP	**0.021**	**0.043**	**0.007**	**0.014**	**0.010**	**0.020**

Table 5. Infratec Soybeans Moisture Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200203	0.36	SECV	0.36	0.72	0.12	0.24	0.17	0.34
Base Calibration + Temp +Stabilization	CN200203	0.34	SECV	0.35	0.70	0.12	0.23	0.16	0.33
Original Calibration 2004	CN20030X	0.37	SECV	0.36	0.71	0.12	0.23	0.17	0.33
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.37	SECV	0.36	0.72	0.12	0.24	0.17	0.34
Validation 553075 (Crop 2004)	CN20030X	0.46	SEP	0.38	0.76	0.13	0.25	0.18	0.35
Validation 1241350 (Crop 2004)	CN20030X	0.45	SEP	**0.39**	**0.78**	**0.13**	**0.26**	**0.18**	**0.37**

Table 6. Infratec Soybeans Protein Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200203	0.60	SECV	0.60	1.19	0.20	0.39	0.28	0.55
Base Calibration + Temp +Stabilization	CN200203	0.57	SECV	0.58	1.17	0.19	0.39	0.27	0.54
Original Calibration 2004	CN20030X	0.62	SECV	0.60	1.19	0.20	0.39	0.28	0.56
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.61	SECV	0.60	1.20	0.20	0.40	0.28	0.56
Validation 553075 (Crop 2004)	CN20030X	0.50	SEP	0.58	1.16	0.19	0.38	0.27	0.54
Validation 1241350 (Crop 2004)	CN20030X	0.48	SEP	**0.56**	**1.13**	**0.19**	**0.37**	**0.26**	**0.53**

Table 7. Infratec Soybeans Oil Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200203	0.49	SECV	0.49	0.98	0.16	0.32	0.23	0.46
Base Calibration + Temp +Stabilization	CN200203	0.47	SECV	0.48	0.96	0.16	0.32	0.22	0.45
Original Calibration 2004	CN20030X	0.52	SECV	0.49	0.98	0.16	0.32	0.23	0.46
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.49	SECV	0.49	0.98	0.16	0.32	0.23	0.46
Validation 553075 (Crop 2004)	CN20030X	0.43	SEP	0.48	0.96	0.16	0.32	0.22	0.45
Validation 1241350 (Crop 2004)	CN20030X	0.40	SEP	**0.47**	**0.93**	**0.15**	**0.31**	**0.22**	**0.43**

Table 8. Infratec Soybeans Fiber Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN200203	0.10	SECV	0.10	0.20	0.03	0.06	0.05	0.09
Base Calibration + Temp +Stabilization	CN200203	0.09	SECV	0.09	0.18	0.03	0.06	0.04	0.09
Original Calibration 2004	CN20030X	0.10	SECV	0.09	0.19	0.03	0.06	0.04	0.09
Base Cal + Temp +Stabilization (Val 2004)	CN20030X	0.09	SECV	**0.09**	0.19	**0.03**	**0.06**	**0.04**	**0.09**
Validation 553075 (Crop 2004)	CN20030X		SEP		0.00				
Validation 1241350 (Crop 2004)	CN20030X		SEP		**0.00**				

Table 9. **Bruins Corn Moisture Tolerance Calculation**

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN01	0.68	SECV	0.68	1.36	0.22	0.45	0.32	0.63
Base Calibration + Temp +Stabilization	CN01	0.64	SECV	0.66	1.32	0.22	0.44	0.31	0.62
2002 Crop Validation	CN01	0.48	SEP	0.60	1.20	0.20	0.40	0.28	0.56
Base calibration 2004		0.67	SECV	0.62	1.24	0.20	0.41	0.29	0.58
Base Calibration + Temp +Stabilization 2004		0.63	SECV	0.62	1.24	0.20	0.41	0.29	0.58
Independent Val/NTEP 2004 (304157/158)		0.30	SEP	0.57	1.13	0.19	0.37	0.26	0.53
Base Calibration NTEP Corn	NTEP corn	0.61	SECV	0.57	1.15	0.19	0.38	0.27	0.53
Base Cal + Temp +Stabilization NTEP Corn	NTEP corn	0.46	SECV	0.56	1.12	0.18	0.37	0.26	0.52
Independent Val NTEP corn (106110/6118)	NTEP corn	0.58	SEP	**0.56**	**1.12**	**0.19**	**0.37**	**0.26**	**0.52**

Table 10. Bruins Corn Protein Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN01	0.57	SECV	0.57	1.14	0.19	0.38	0.27	0.53
Base Calibration + Temp +Stabilization	CN01	0.42	SECV	0.50	0.99	0.16	0.33	0.23	0.46
2002 Crop Validation	CN01	0.46	SEP	0.48	0.97	0.16	0.32	0.23	0.45
Base calibration 2004		0.43	SECV	0.47	0.94	0.16	0.31	0.22	0.44
Base Calibration + Temp +Stabilization 2004		0.44	SECV	0.46	0.93	0.15	0.31	0.22	0.43
Independent Val/NTEP 2004 (304157/158)		0.31	SEP	0.44	0.88	0.15	0.29	0.21	0.41
Base Calibration NTEP Corn	NTEP corn	0.39	SECV	0.43	0.86	0.14	0.28	0.20	0.40
Base Cal + Temp +Stabilization NTEP Corn	NTEP corn	0.34	SECV	0.42	0.84	0.14	0.28	0.20	0.39
Independent Val NTEP corn (106110/6118)	NTEP corn	0.42	SEP	**0.42**	**0.84**	**0.14**	**0.28**	**0.20**	**0.39**

Table 11. Bruins Corn Oil Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN01	0.50	SECV	0.50	1.00	0.17	0.33	0.23	0.47
Base Calibration + Temp +Stabilization	CN01	0.41	SECV	0.46	0.91	0.15	0.30	0.21	0.42
2002 Crop Validation	CN01	0.35	SEP	0.42	0.84	0.14	0.28	0.20	0.39
Base calibration 2004		0.47	SECV	0.43	0.87	0.14	0.29	0.20	0.40
Base Calibration + Temp +Stabilization 2004		0.43	SECV	0.43	0.86	0.14	0.29	0.20	0.40
Independent Val/NTEP 2004 (304157/158)		0.40	SEP	0.43	0.85	0.14	0.28	0.20	0.40
Base Calibration NTEP Corn	NTEP corn	0.34	SECV	0.41	0.83	0.14	0.27	0.19	0.39
Base Cal + Temp +Stabilization NTEP Corn	NTEP corn	0.31	SECV	0.4	0.80	0.13	0.26	0.19	0.37
Independent Val NTEP corn (106110/6118)	NTEP corn	0.49	SEP	**0.41**	**0.82**	**0.14**	**0.27**	**0.19**	**0.38**

Table 12. Bruins Corn Starch Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN01	0.86	SECV	0.86	1.72	0.28	0.57	0.40	0.80
Base Calibration + Temp +Stabilization	CN01	0.68	SECV	0.77	1.54	0.25	0.51	0.36	0.72
2002 Crop Validation	CN01	0.63	SEP	0.72	1.45	0.24	0.48	0.34	0.68
Base calibration 2004			SECV	0.72	1.45	0.24	0.48	0.34	0.68
Base Calibration + Temp +Stabilization 2004			SECV	0.72	1.45	0.24	0.48	0.34	0.68
Independent Val/NTEP 2004 (304157/158)			SEP	0.72	1.45	0.24	0.48	0.34	0.68
Base Calibration NTEP Corn	NTEP corn	0.59	SECV	0.69	1.38	0.23	0.46	0.32	0.64
Base Cal + Temp +Stabilization NTEP Corn	NTEP corn	0.50	SECV	0.65	1.30	0.22	0.43	0.30	0.61
Independent Val NTEP corn (106110/6118)	NTEP corn	0.73	SEP	**0.67**	**1.33**	**0.22**	**0.44**	**0.31**	**0.62**

Table 13. Bruins Corn Density Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	CN01	0.017	SECV	0.017	0.034	0.006	0.011	0.008	0.016
Base Calibration + Temp +Stabilization	CN01	0.011	SECV	0.014	0.028	0.005	0.009	0.007	0.013
2002 Crop Validation	CN01	0.018	SEP	0.015	0.031	0.005	0.010	0.007	0.014
Base calibration 2004			SECV	0.015	0.031	0.005	0.010	0.007	0.014
Base Calibration + Temp +Stabilization 2004			SECV	0.015	0.031	0.005	0.010	0.007	0.014
Independent Val/NTEP 2004 (304157/158)			SEP	0.015	0.031	0.005	0.010	0.007	0.014
Base Calibration NTEP Corn	NTEP corn	0.015	SECV	0.015	0.031	0.005	0.010	0.007	0.014
Base Cal + Temp +Stabilization NTEP Corn	NTEP corn	0.012	SECV	0.015	0.029	0.005	0.010	0.007	0.014
Independent Val NTEP corn (106110/6118)	NTEP corn	0.024	SEP	**0.016**	**0.032**	**0.005**	**0.011**	**0.008**	**0.015**

Table 14. Bruins Soybeans Moisture Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	Soybeans02	0.33	SECV	0.33	0.66	0.11	0.22	0.15	0.31
Base Calibration + Temp +Stabilization	Soybeans02	0.32	SECV	0.32	0.65	0.11	0.21	0.15	0.30
2002 Crop Validation (106110/6118)	Soybeans02	0.45	SEP	0.37	0.73	0.12	0.24	0.17	0.34
2003 Crop Validation	Soybeans02	0.40	SEP	0.38	0.75	0.12	0.25	0.18	0.35
Base Calibration NTEP Corn	NTEP2005 sb	0.34	SECV	0.37	0.74	0.12	0.24	0.17	0.34
Base Cal + Temp +Stabilization NTEP Sb	NTEP2005 sb	0.34	SECV	0.36	0.73	0.12	0.24	0.17	0.34
Independent Val NTEP sb (106110/6118)	NTEP2005 sb	0.55	SEP	**0.39**	**0.78**	**0.13**	**0.26**	**0.18**	**0.36**

Table 15. Bruins Soybeans Protein Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	Soybeans02	0.58	SECV	0.58	1.16	0.19	0.38	0.27	0.54
Base Calibration + Temp +Stabilization	Soybeans02	0.59	SECV	0.58	1.17	0.19	0.39	0.27	0.54
2002 Crop Validation (106110/6118)	Soybeans02	0.58	SEP	0.58	1.16	0.19	0.38	0.27	0.54
2003 Crop Validation	Soybeans02	0.60	SEP	0.59	1.17	0.19	0.39	0.27	0.55
Base Calibration NTEP Corn	NTEP2005 sb	0.45	SECV	0.56	1.12	0.19	0.37	0.26	0.52
Base Cal + Temp +Stabilization NTEP Sb	NTEP2005 sb	0.46	SECV	0.54	1.09	0.18	0.36	0.25	0.51
Independent Val NTEP sb (106110/6118)	NTEP2005 sb	0.60	SEP	**0.55**	**1.10**	**0.18**	**0.36**	**0.26**	**0.51**

Table16. Bruins Soybeans Oil Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	Soybeans02	0.49	SECV	0.49	0.98	0.16	0.32	0.23	0.46
Base Calibration + Temp +Stabilization	Soybeans02	0.45	SECV	0.47	0.94	0.16	0.31	0.22	0.44
2002 Crop Validation (106110/6118)	Soybeans02	0.41	SEP	0.45	0.90	0.15	0.30	0.21	0.42
2003 Crop Validation	Soybeans02	0.50	SEP	0.46	0.93	0.15	0.31	0.22	0.43
Base Calibration NTEP Corn	NTEP2005 sb	0.35	SECV	0.44	0.88	0.15	0.29	0.21	0.41
Base Cal + Temp +Stabilization NTEP Sb	NTEP2005 sb	0.35	SECV	0.43	0.85	0.14	0.28	0.20	0.40
Independent Val NTEP sb (106110/6118)	NTEP2005 sb	0.44	SEP	**0.43**	**0.86**	**0.14**	**0.28**	**0.20**	**0.40**

Table 17. Bruins Soybeans Fiber Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration	Soybeans02	0.07	SECV	0.07	0.14	0.02	0.05	0.03	0.07
Base Calibration + Temp +Stabilization	Soybeans02	0.08	SECV	0.08	0.15	0.03	0.05	0.03	0.07
2002 Crop Validation (106110/6118)	Soybeans02	0.07	SEP	0.07	0.15	0.02	0.05	0.03	0.07
2003 Crop Validation	Soybeans02			0.07	0.15	0.02	0.05	0.03	0.07
Base Calibration NTEP Corn	NTEP2005 sb	0.07	SECV	0.07	0.15	0.02	0.05	0.03	0.07
Base Cal + Temp +Stabilization NTEP sb	NTEP2005 sb	0.07	SECV	0.07	0.14	0.02	0.05	0.03	0.07
Independent Val NTEP sb (106110/6118)	NTEP2005 sb			**0.07**	**0.14**	**0.02**	**0.05**	**0.03**	**0.07**

Table 18. Perten DA 7200 Corn Moisture Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.35	SECV	0.35	0.70	0.12	0.23	0.16	0.33
Base Calibration + Temp +Stabilization		0.33	SECV	**0.34**	**0.68**	**0.11**	**0.22**	**0.16**	**0.32**

Table 19. Perten DA 7200 Corn Protein Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.41	SECV	0.41	0.82	0.14	0.27	0.19	0.38
Base Calibration + Temp +Stabilization		0.42	SECV	**0.42**	**0.83**	**0.14**	**0.27**	**0.19**	**0.39**

Table 20. Perten DA 7200 Corn Oil Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.48	SECV	0.48	0.96	0.16	0.32	0.22	0.45
Base Calibration + Temp +Stabilization		0.33	SECV	**0.41**	**0.81**	**0.13**	**0.27**	**0.19**	**0.38**

Table 21. Perten DA 7200 Corn Starch Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.76	SECV	0.76	1.52	0.25	0.50	0.36	0.71
Base Calibration + Temp +Stabilization		0.80	SECV	**0.78**	**1.56**	**0.26**	**0.51**	**0.36**	**0.73**

Table 22. Perten DA 7200 Corn Density Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.014	SECV	0.014	0.03	0.005	0.009	0.007	0.013
Base Calibration + Temp +Stabilization		0.010	SECV	**0.012**	**0.02**	**0.004**	**0.008**	**0.006**	**0.011**

Table 23. Perten DA 7200 Soybean Moisture Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.52	SECV	0.52	1.04	0.17	0.34	0.24	0.49
Independent Validation		0.34	SEP	**0.43**	**0.86**	**0.14**	**0.28**	**0.20**	**0.40**

Table 24. Perten DA 7200 Soybean Protein Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.78	SECV	0.78	1.56	0.26	0.51	0.36	0.73
Independent Validation		0.72	SEP	**0.75**	**1.50**	**0.25**	**0.50**	**0.35**	**0.70**

Table 25. Perten DA 7200 Soybean Oil Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.54	SECV	0.54	1.08	0.18	0.36	0.25	0.50
Independent Validation		0.34	SEP	**0.44**	**0.88**	**0.15**	**0.29**	**0.21**	**0.41**

Table 25. Perten DA 7200 Soybean Fiber Tolerance Calculation

Item	CAL ID	SEP/ SECV	Type	Cum. Ave	95% CI	0.33* SEP/ SECV	95% CI	SED	95% CI
Base calibration		0.08	SECV	0.08	0.16	0.03	0.05	0.04	0.07
Independent Validation		0.08	SEP	**0.08**	**0.16**	**0.03**	**0.05**	**0.04**	**0.07**

APPENDIX B. NIRS DAILY CHECK SAMPLE

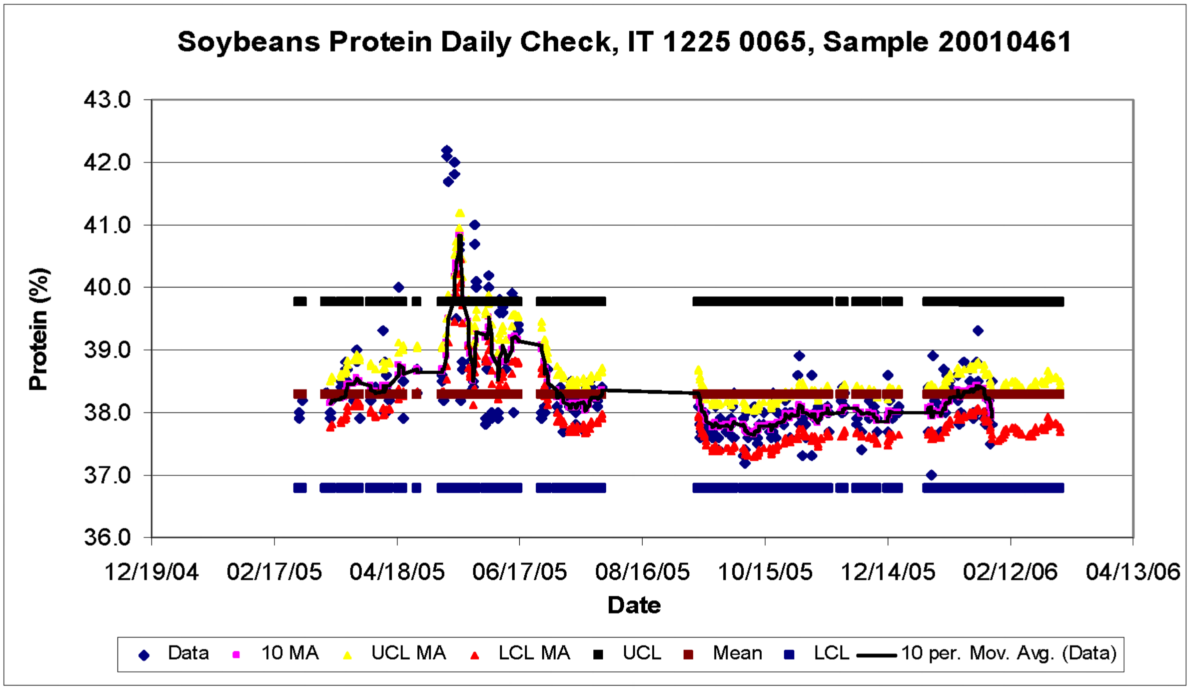


Figure 1. Soybeans Protein Daily Check, Sample 20010461, IT 1225 0065 Control Chart

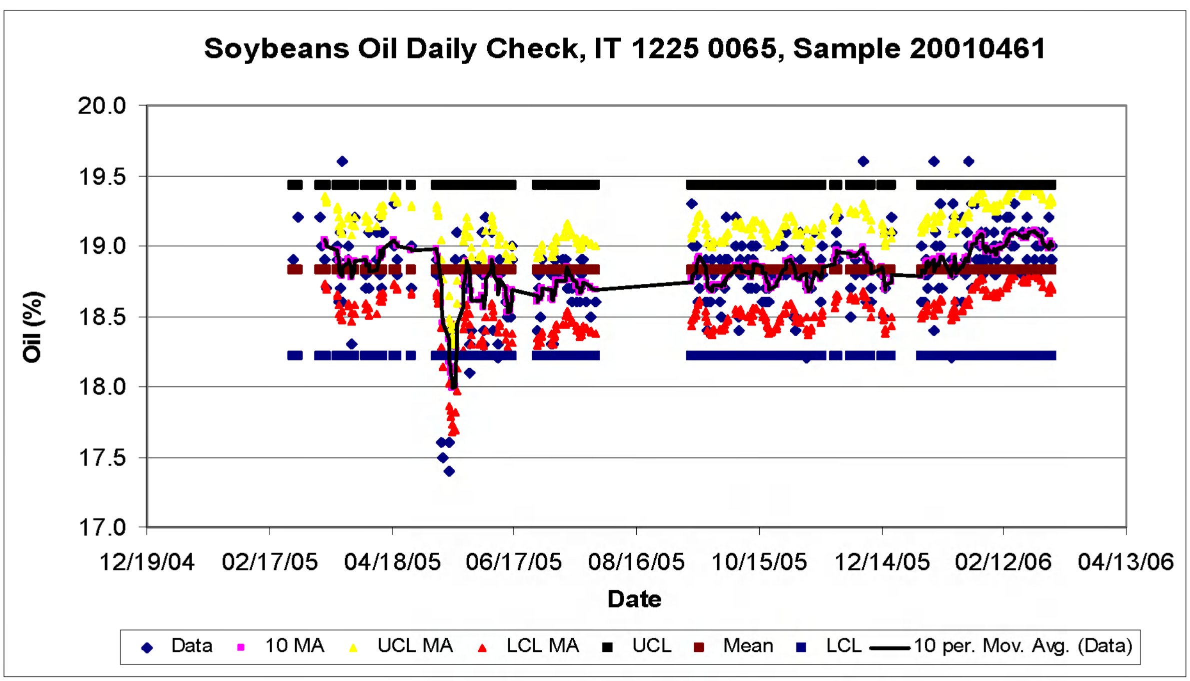


Figure 2. Soybeans Oil Daily Check, Sample 20010461, IT 1225 0065 Control Chart

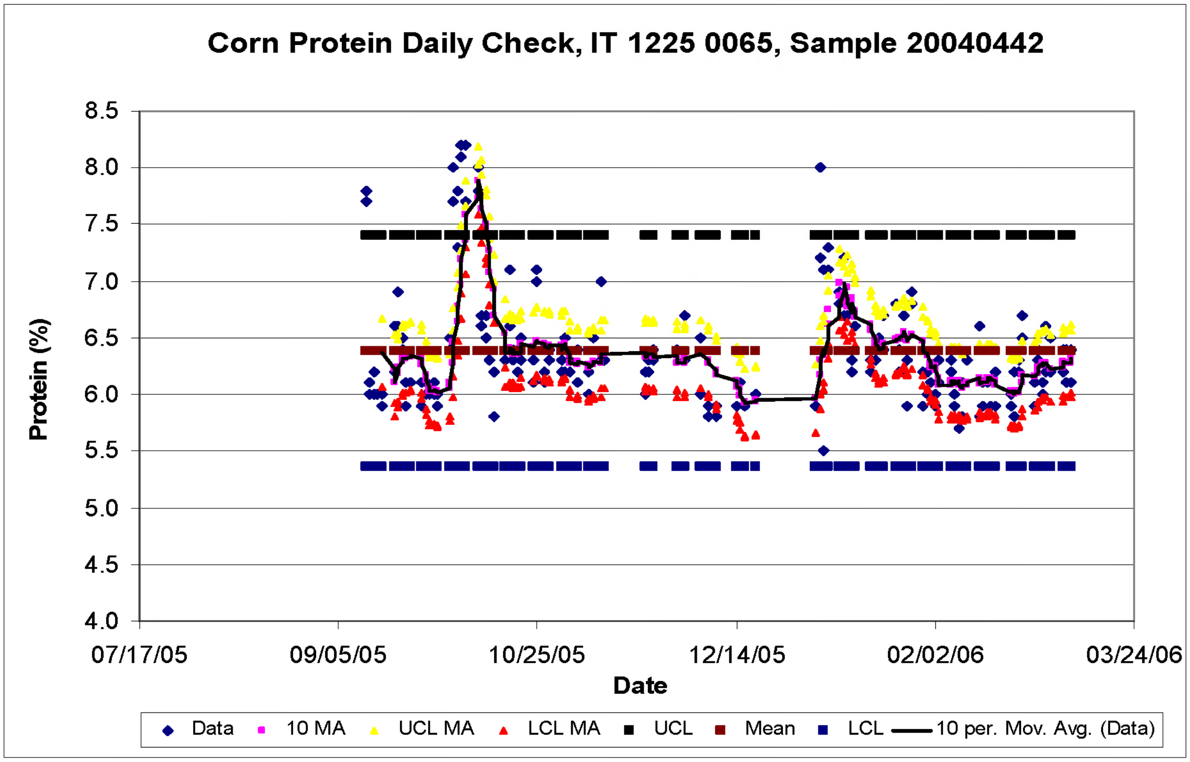


Figure 3. Corn Protein Daily Check, Sample 20040442, IT 1225 0065 Control Chart

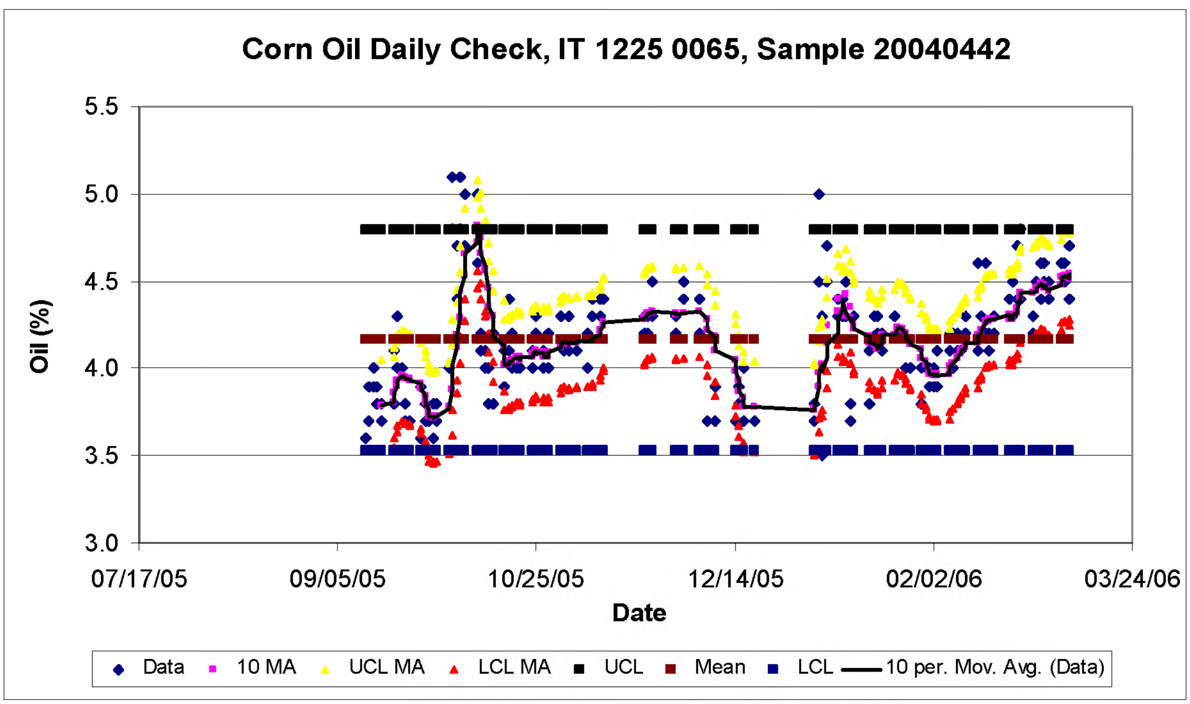


Figure 4. Corn Oil Daily Check, Sample 20040442, IT 1225 0065 Control Chart

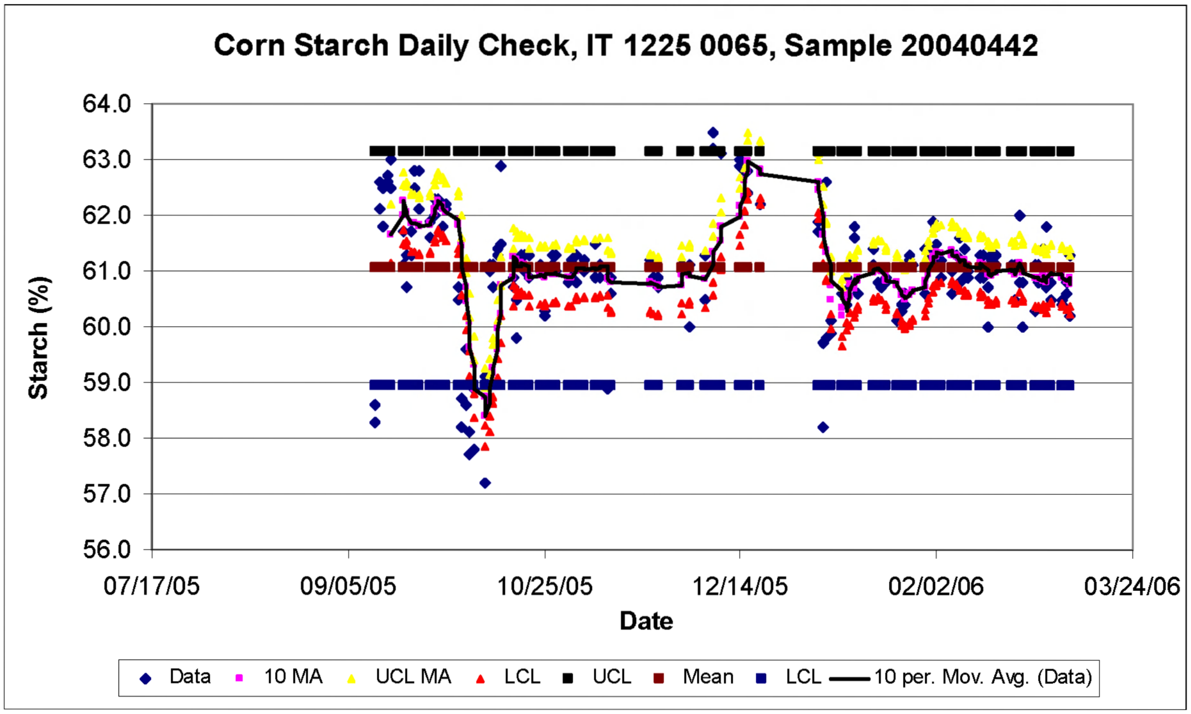


Figure 5. Corn Starch Daily Check, Sample 20040442, IT 1225 0065 Control Chart

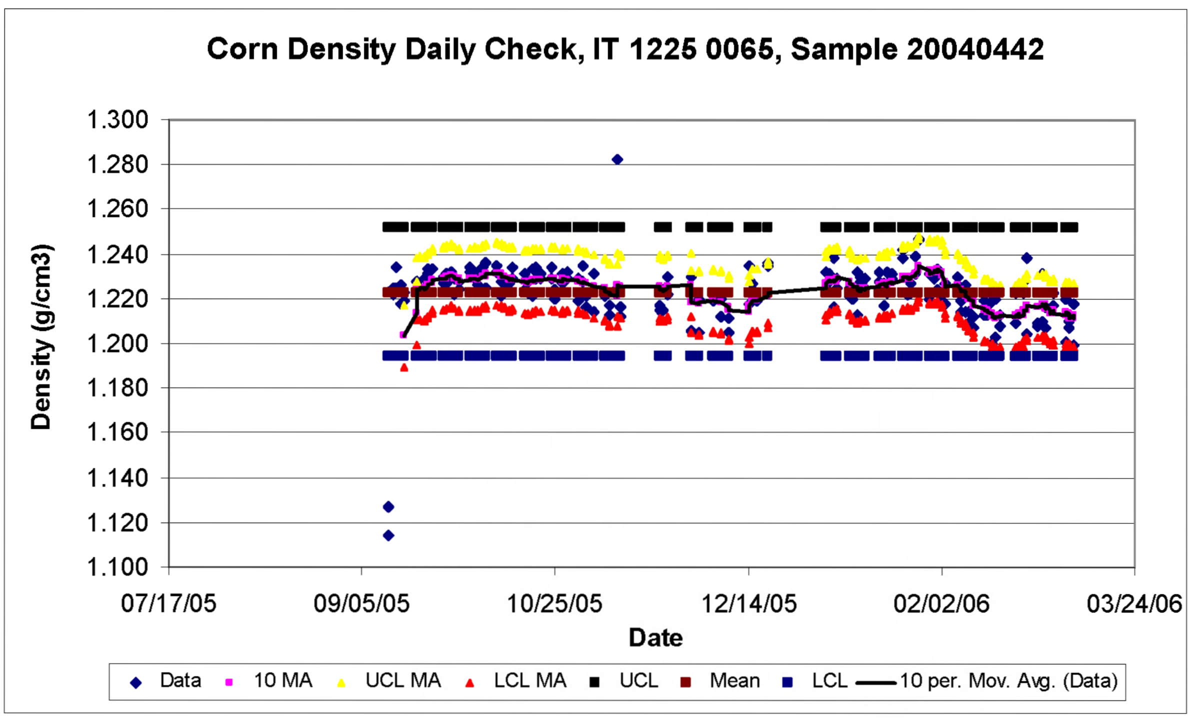


Figure 6. Corn Density Daily Check, Sample 20040442, IT 1225 0065 Control Chart

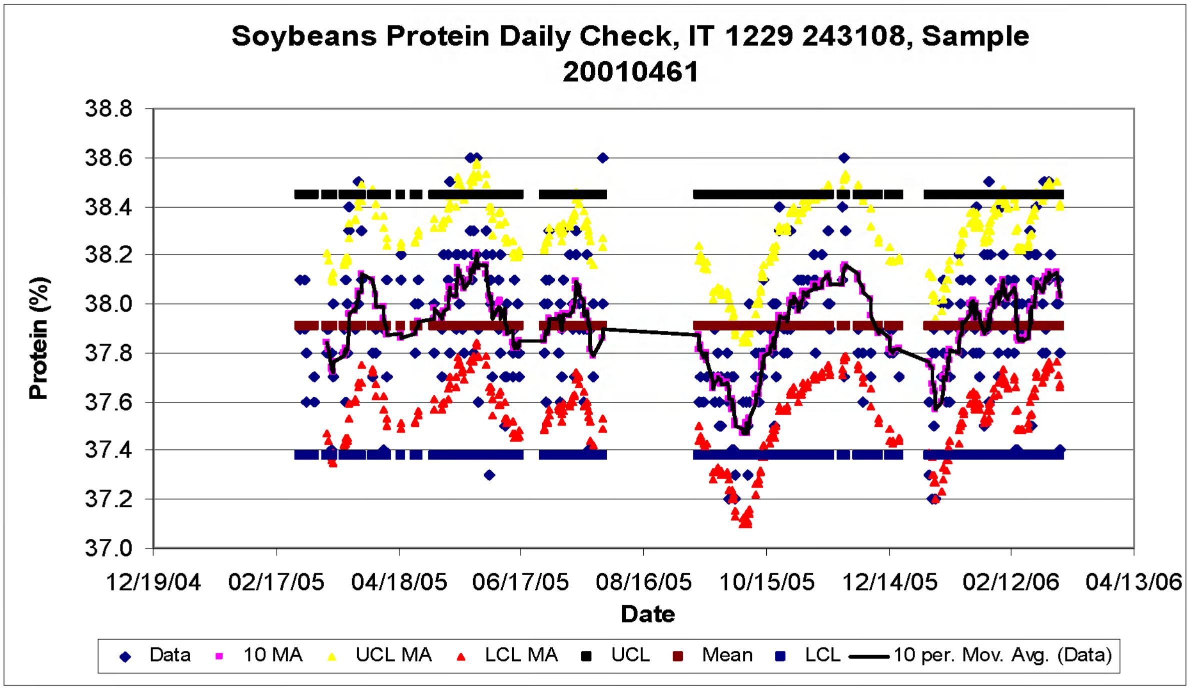


Figure 7. Soybeans Protein Daily Check, Sample 20010461, IT 1229 243108 Control Chart

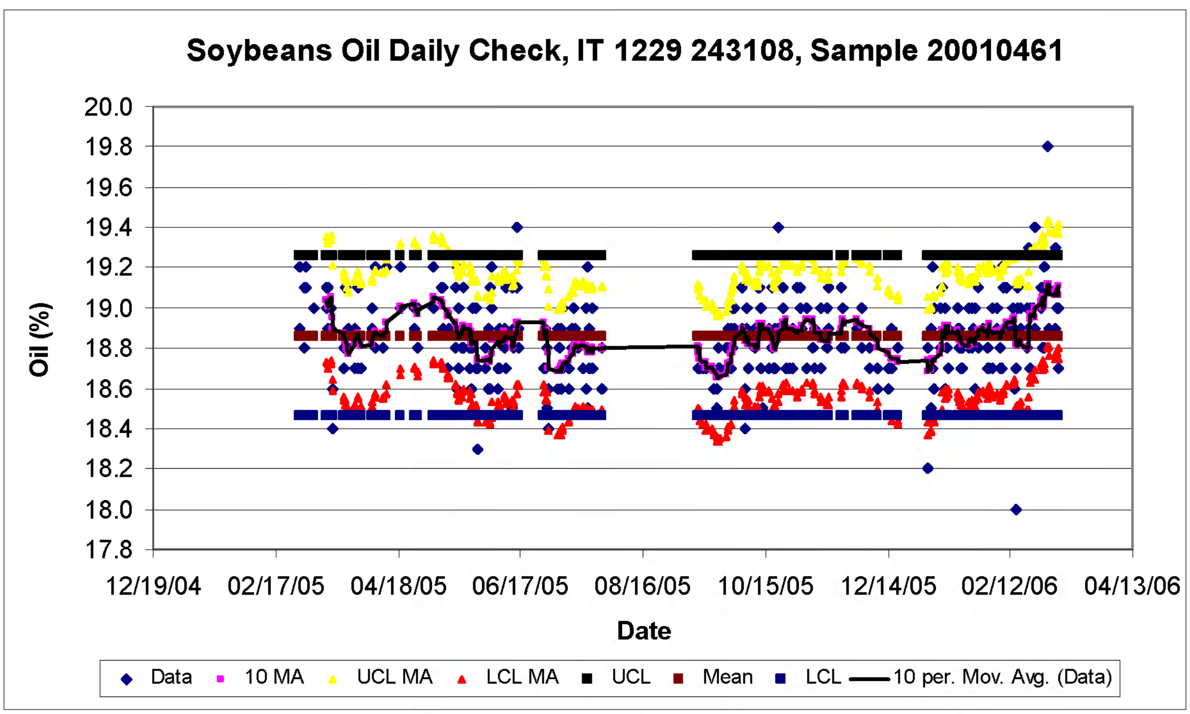


Figure 8. Soybeans Oil Daily Check, Sample 20010461, IT 1229 243108 Control Chart

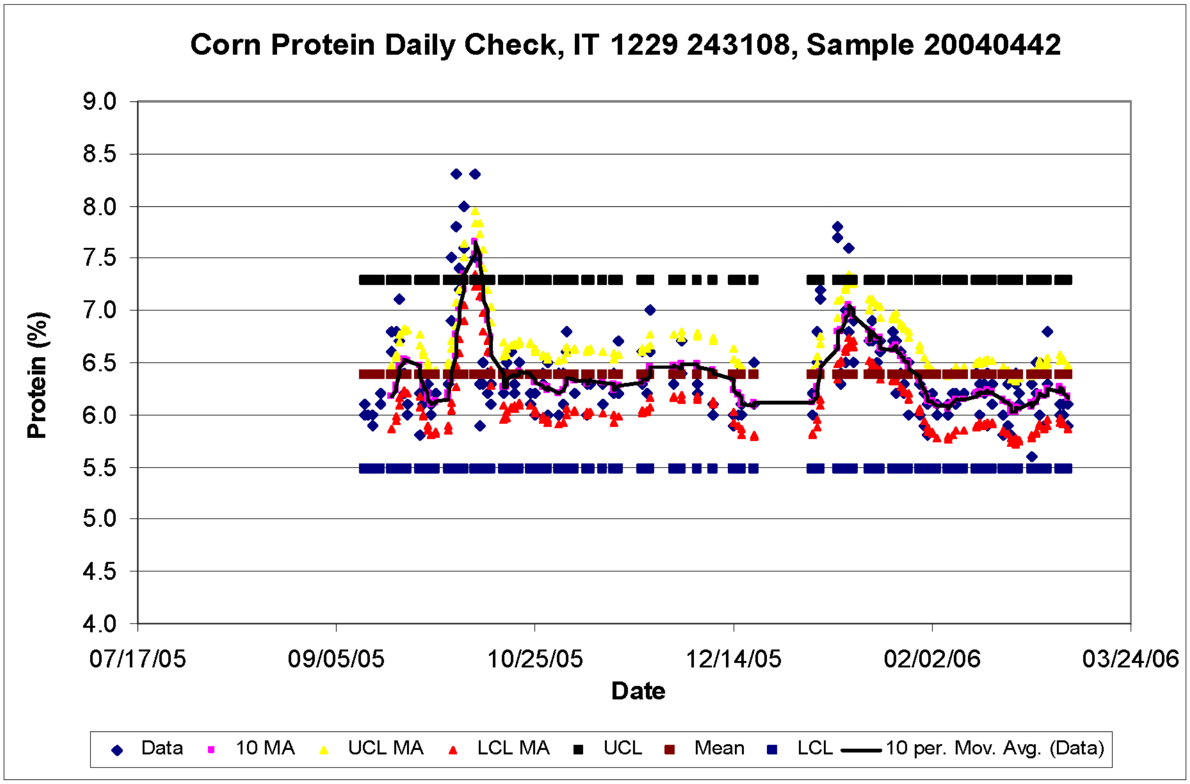


Figure 9. Corn Protein Daily Check, Sample 20040442, IT 1229 243108 Control Chart

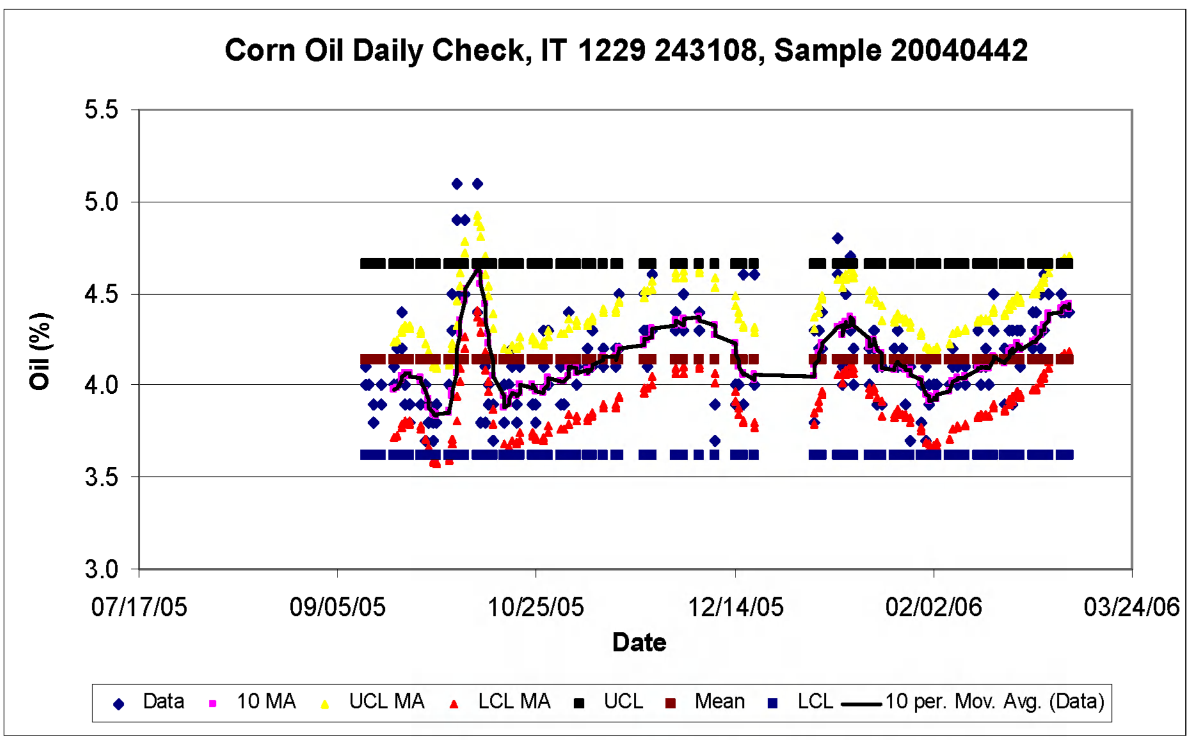


Figure 10. Corn Oil Daily Check, Sample 20040442, IT 1229 243108 Control Chart

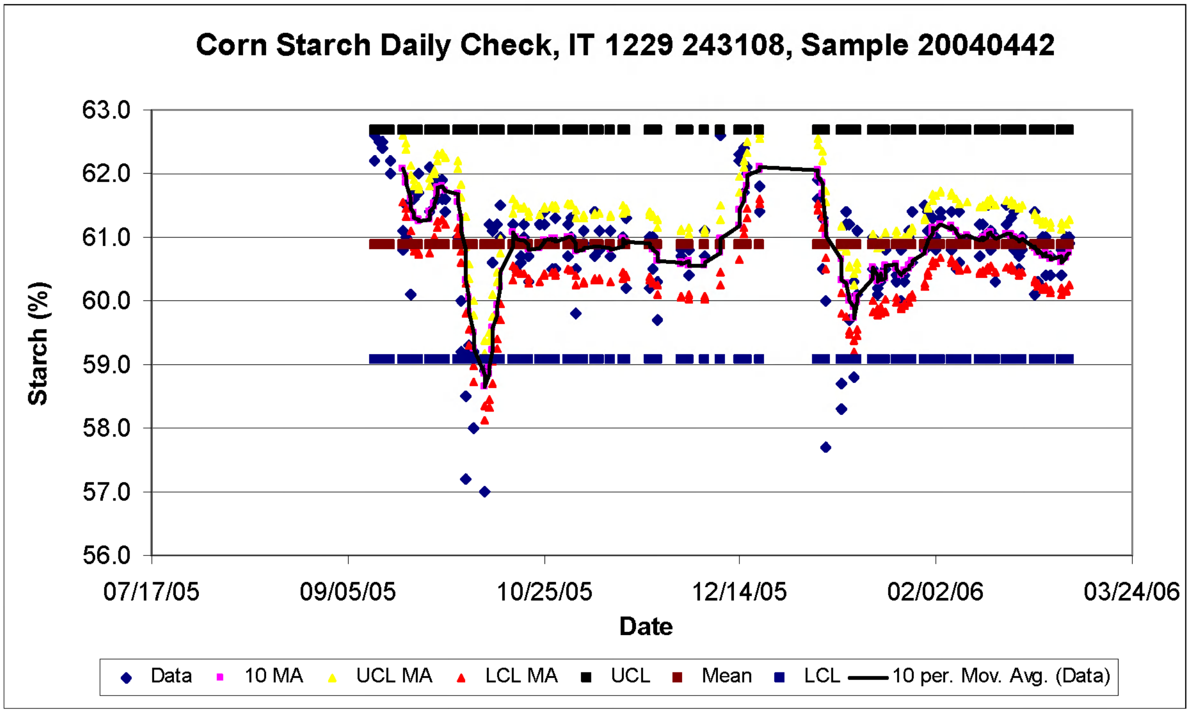


Figure 11. Corn Starch Daily Check, Sample 20040442, IT 1229 243108 Control Chart

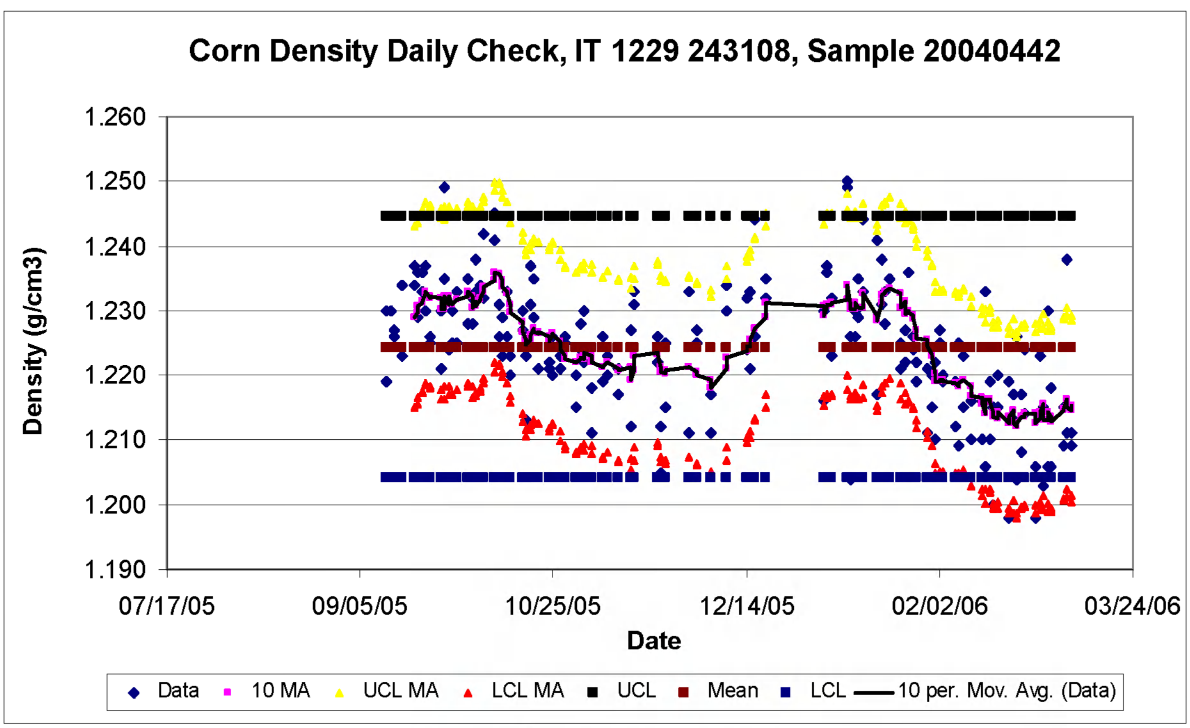


Figure 12. Corn Density Daily Check, Sample 20040442, IT 1229 243108 Control Chart

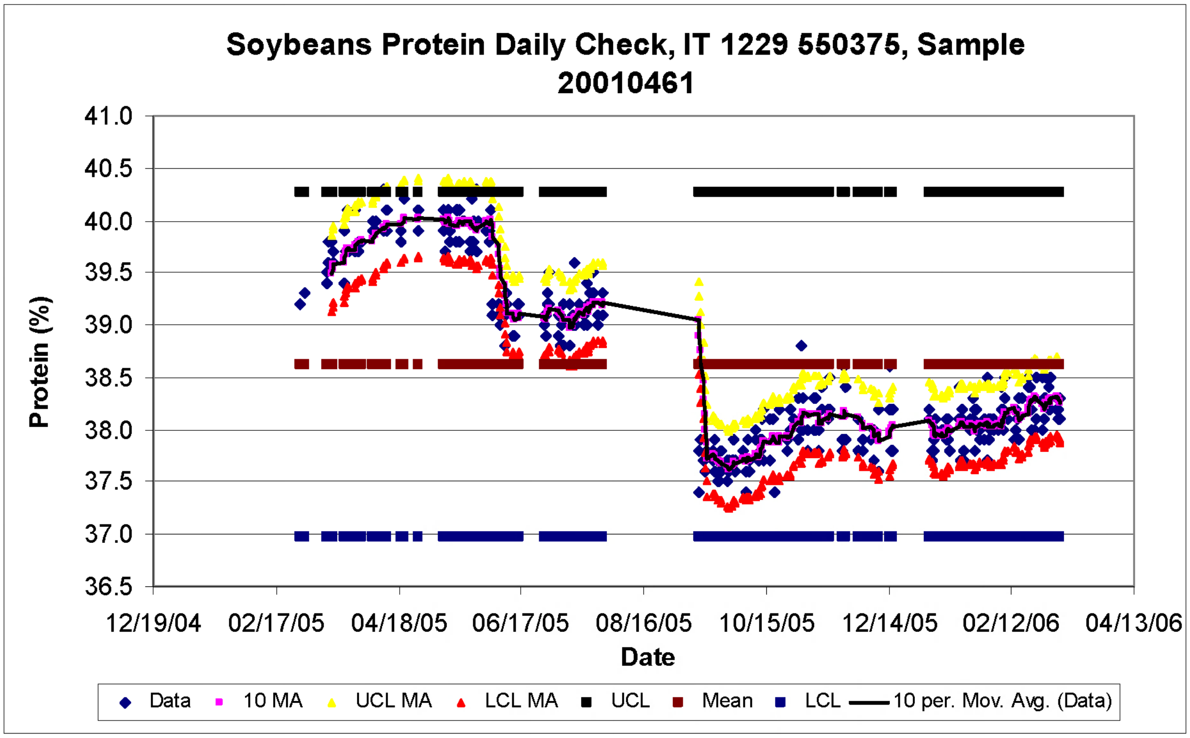


Figure 13. Soybeans Protein Daily Check, Sample 20010461, IT 1229 553075 Control Chart

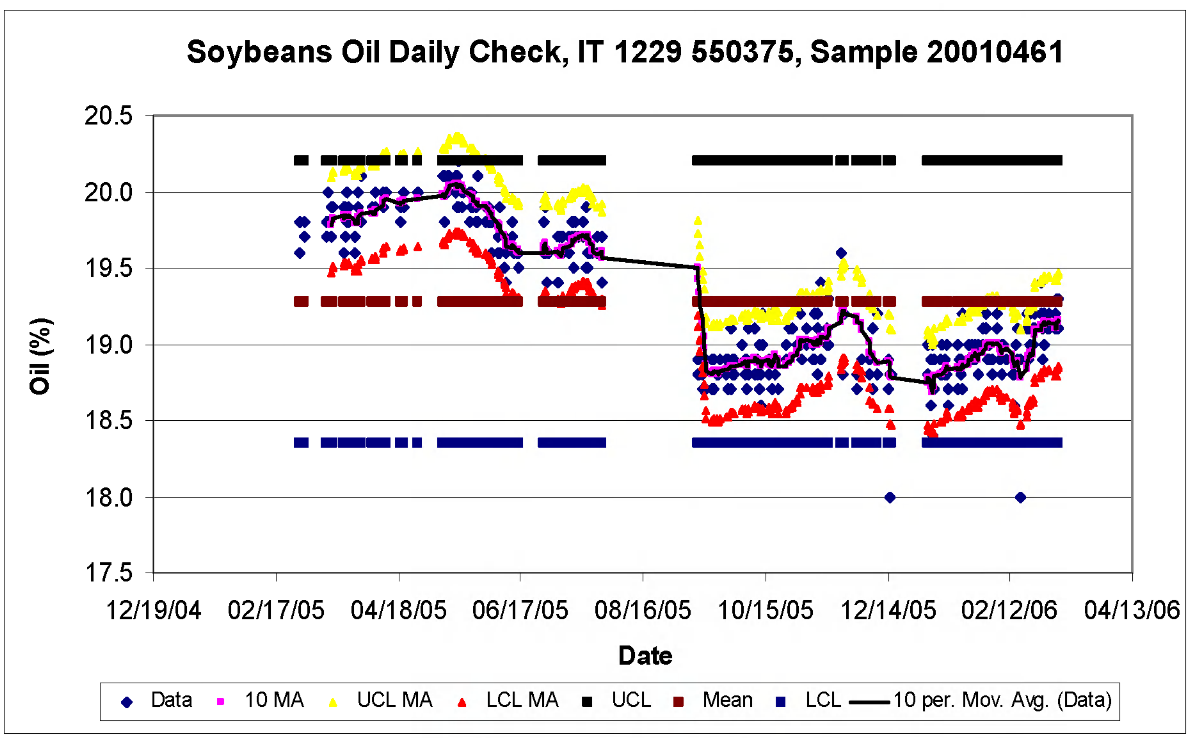


Figure 14. Soybeans Oil Daily Check, Sample 20010461, IT 1229 553075 Control Chart

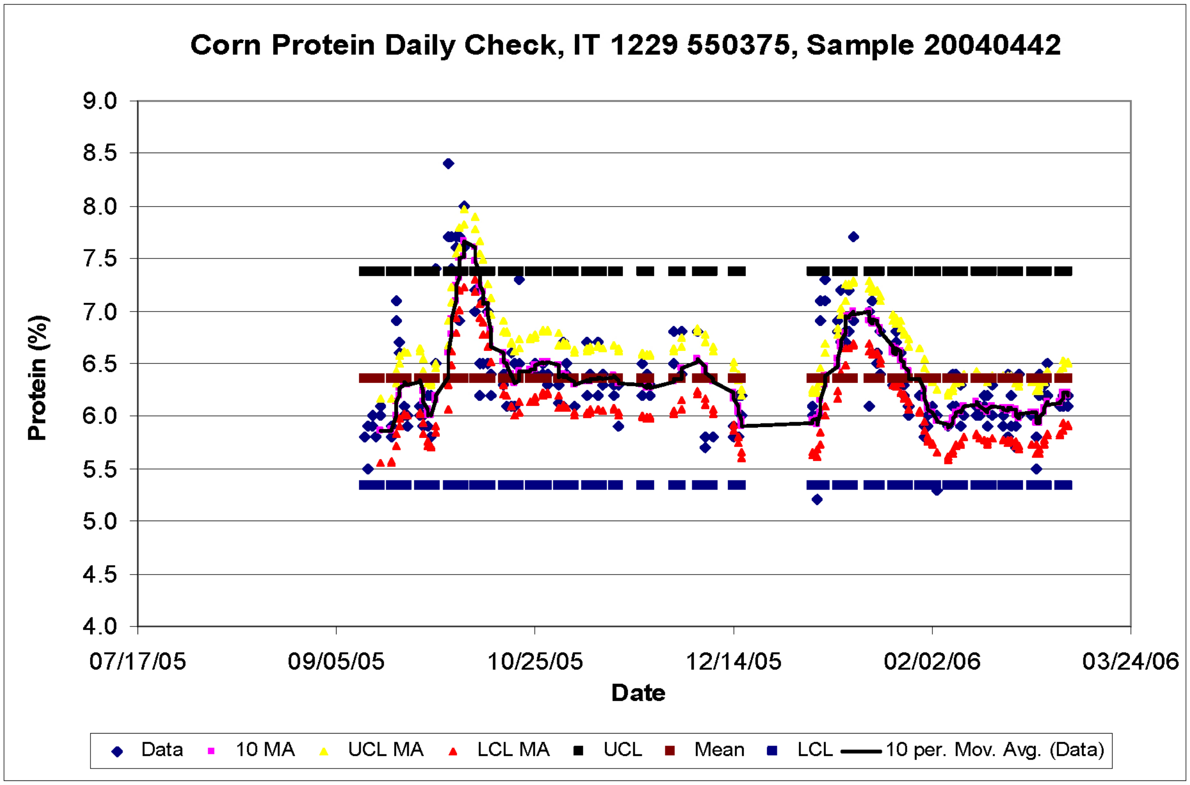


Figure 15. Corn Protein Daily Check, Sample 20040442, IT 1229 553075 Control Chart

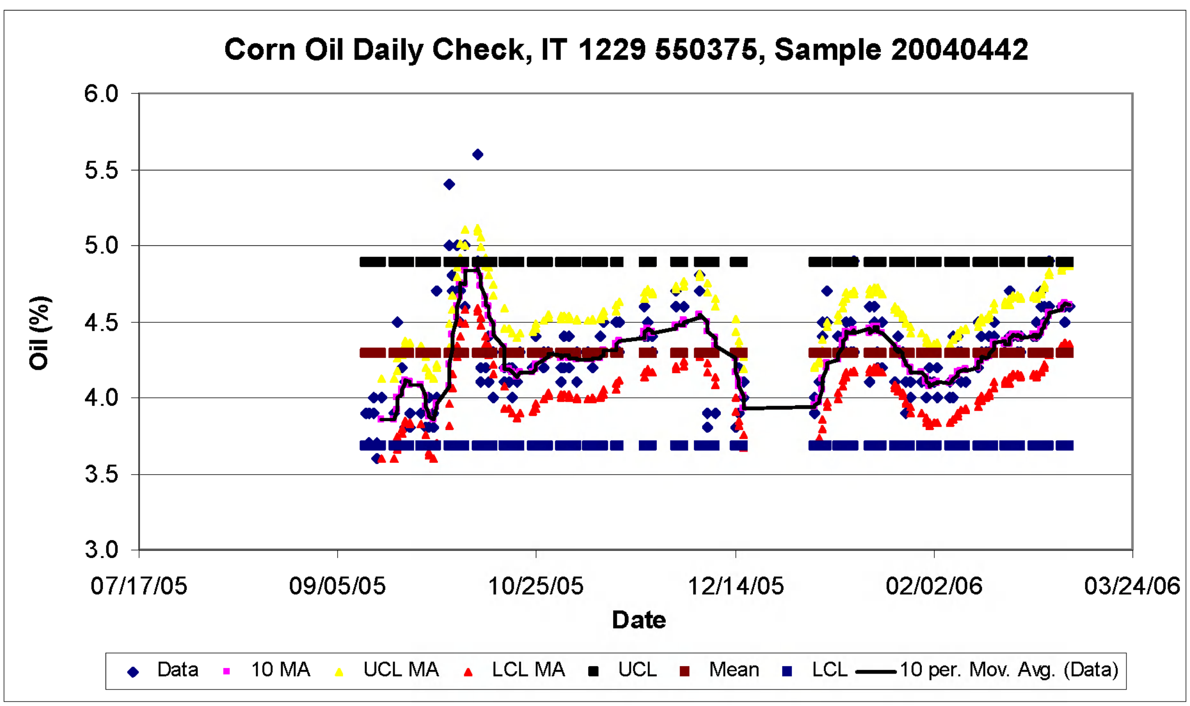


Figure 16. Corn Oil Daily Check, Sample 20040442, IT 1229 553075 Control Chart

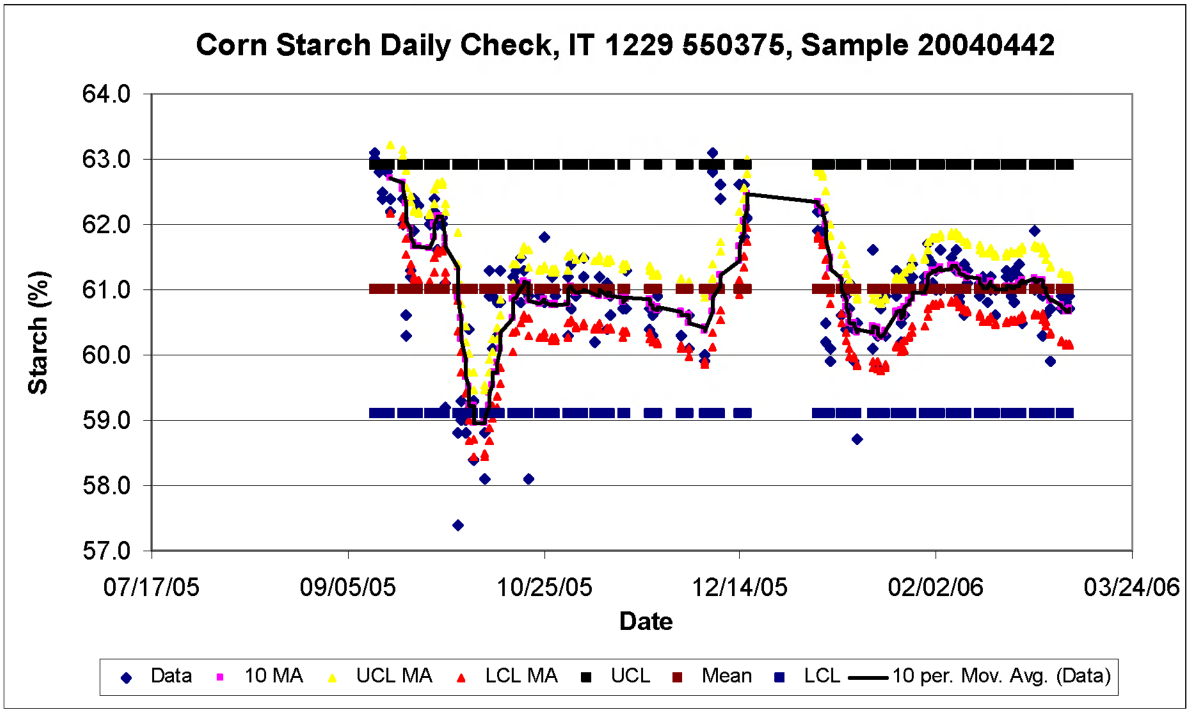


Figure 17. Corn Starch Daily Check, Sample 20040442, IT 1229 553075 Control Chart

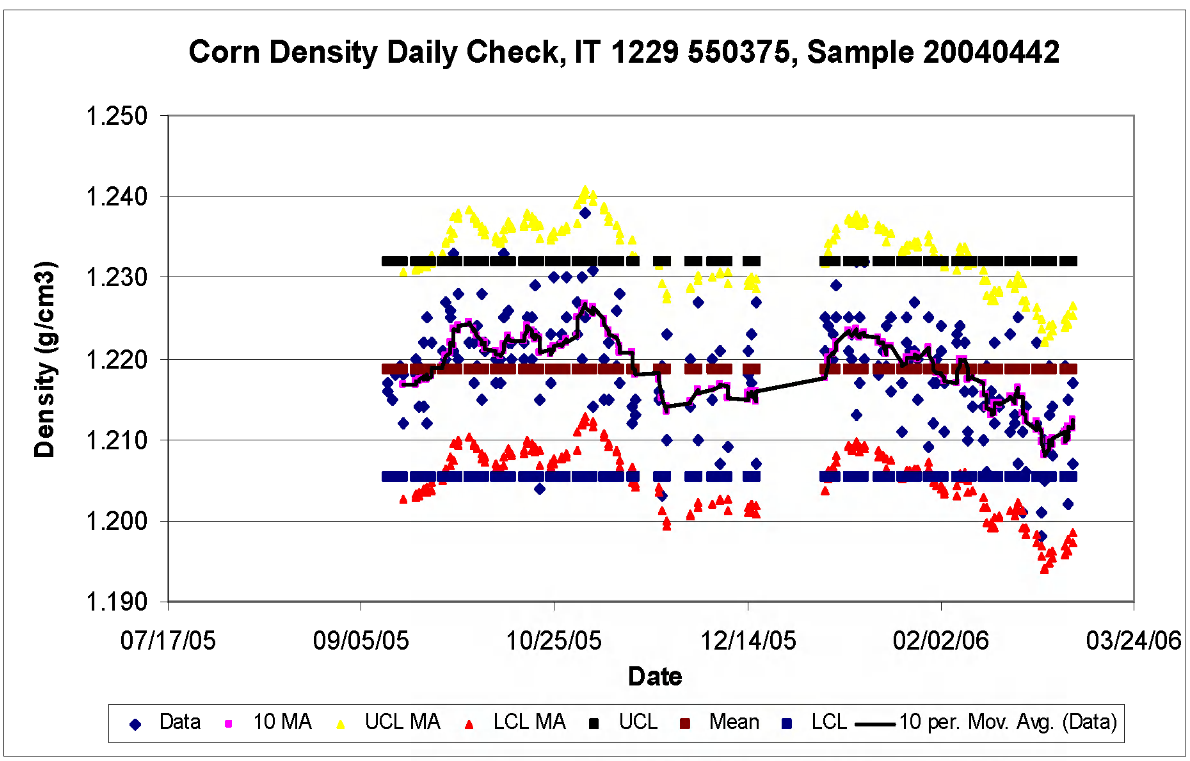


Figure 18. Corn Density Daily Check, Sample 20040442, IT 1229 553075 Control Chart

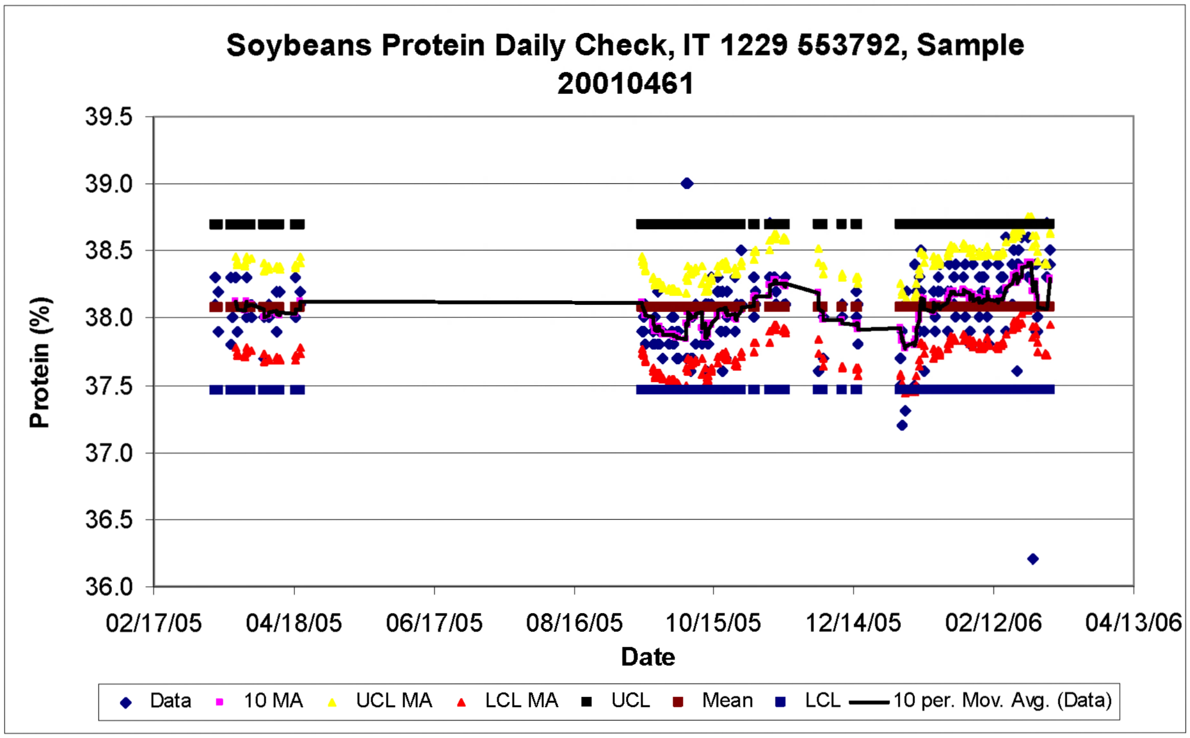


Figure 19. Soybeans Protein Daily Check, Sample 20010461, IT 1229 553792 Control Chart

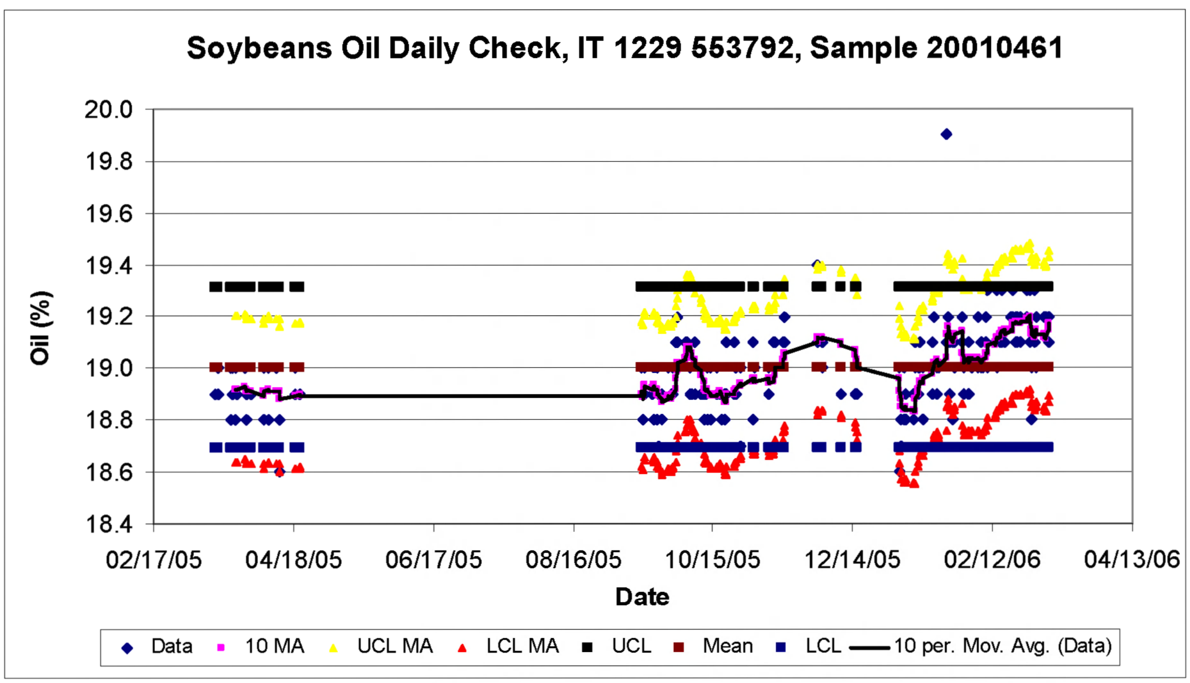


Figure 20. Soybeans Oil Daily Check, Sample 20010461, IT 1229 553792 Control Chart

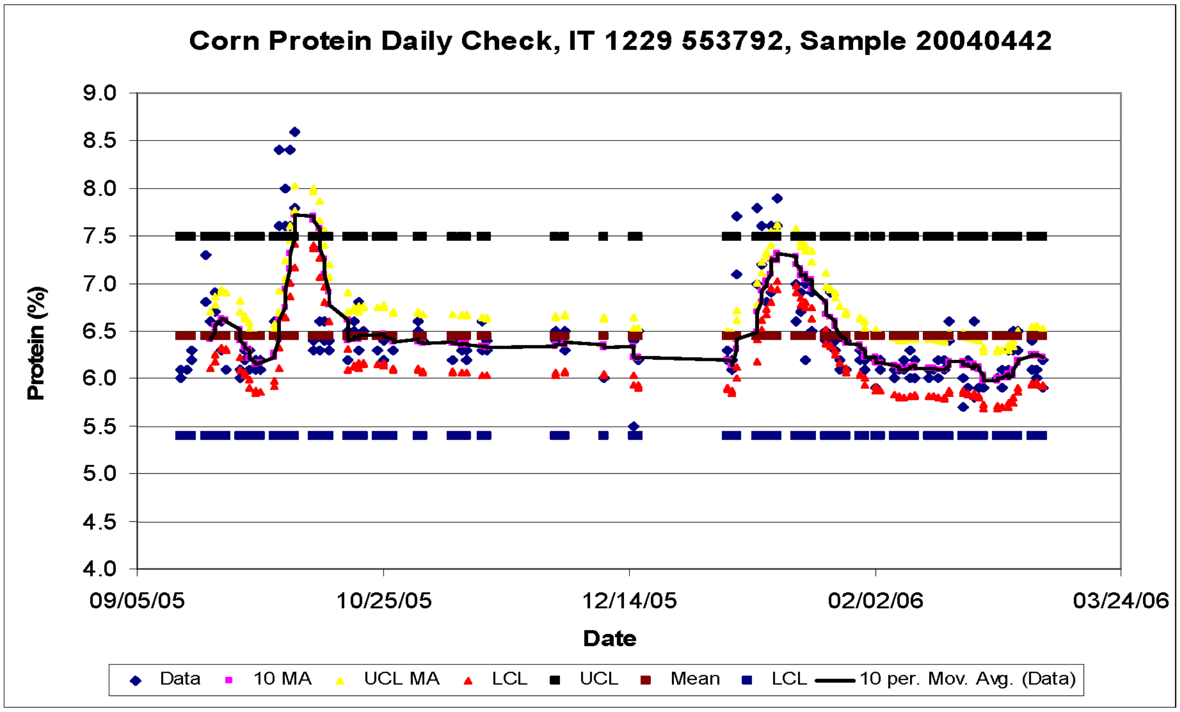


Figure 21. Corn Protein Daily Check, Sample 20040442, IT 1229 553792 Control Chart

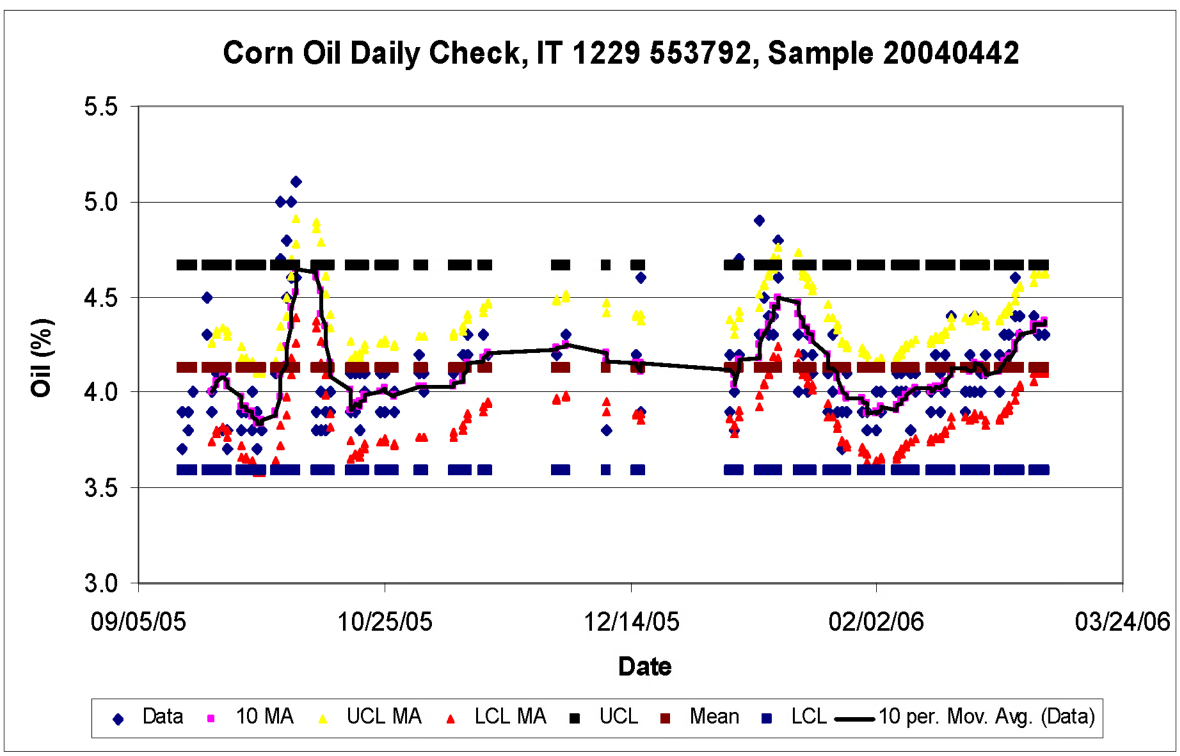


Figure 22. Corn Oil Daily Check, Sample 20040442, IT 1229 553792 Control Chart

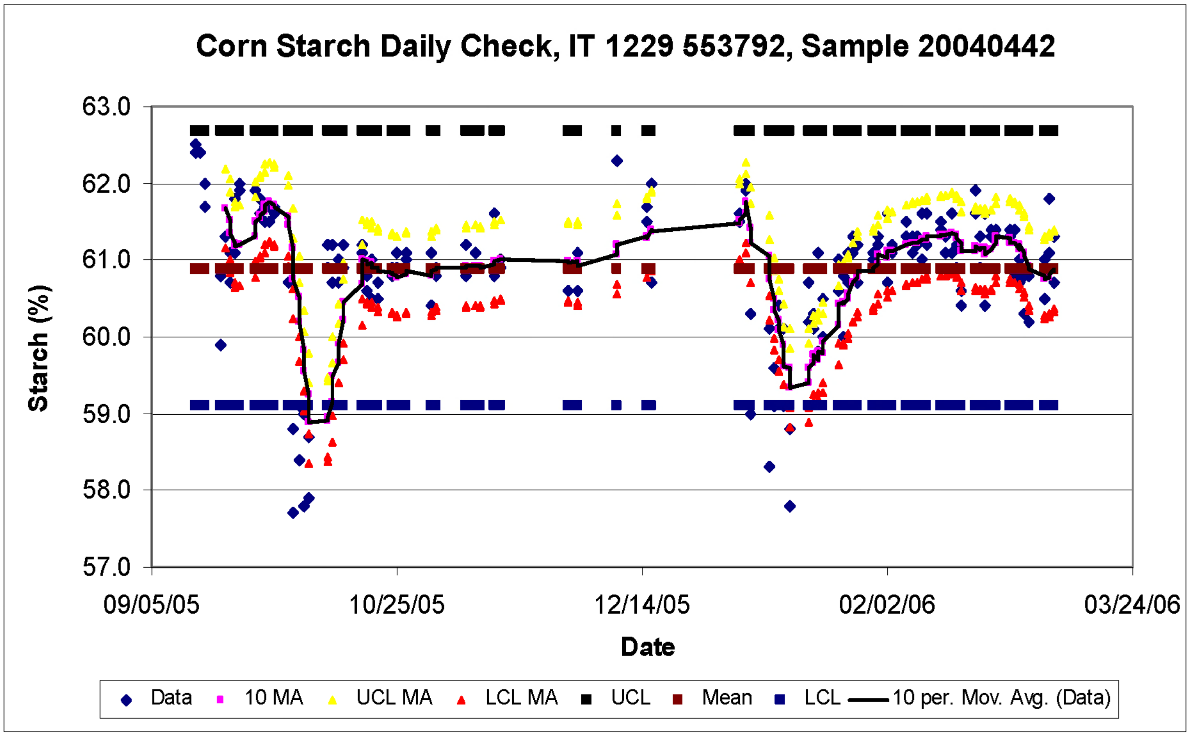


Figure 23. Corn Starch Daily Check, Sample 20040442, IT 1229 553792 Control Chart

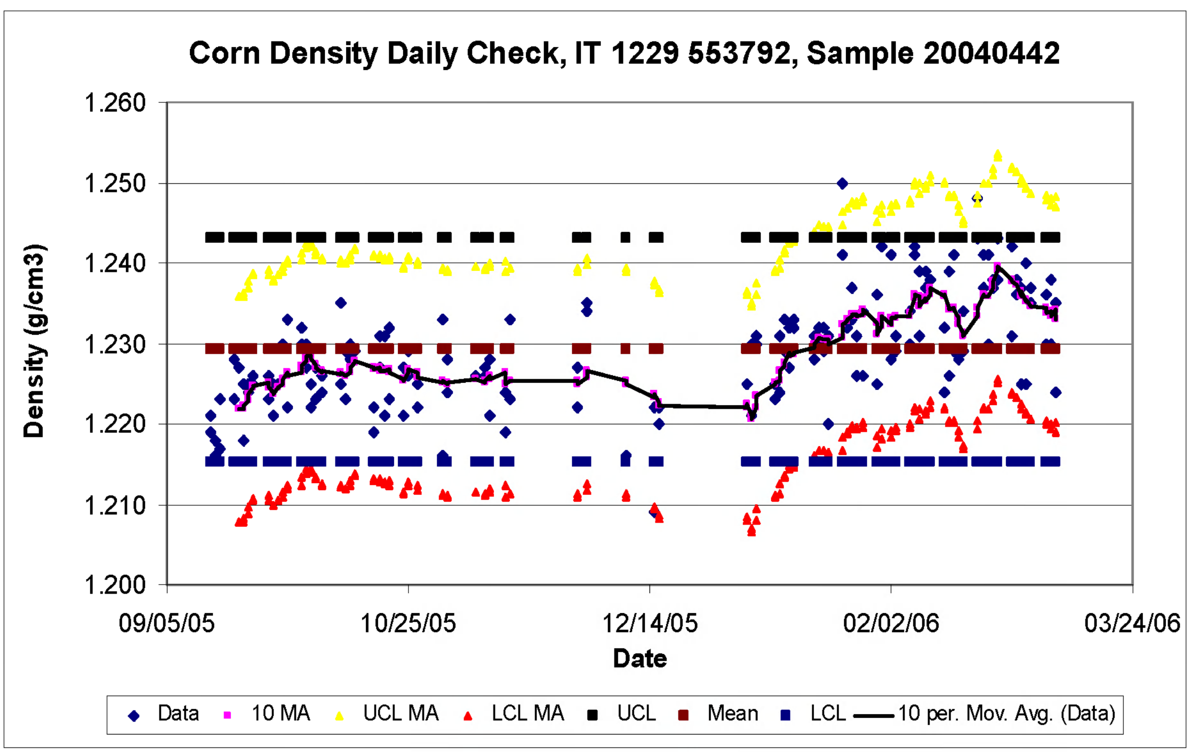


Figure 24. Corn Density Daily Check, Sample 20040442, IT 1229 553792 Control Chart

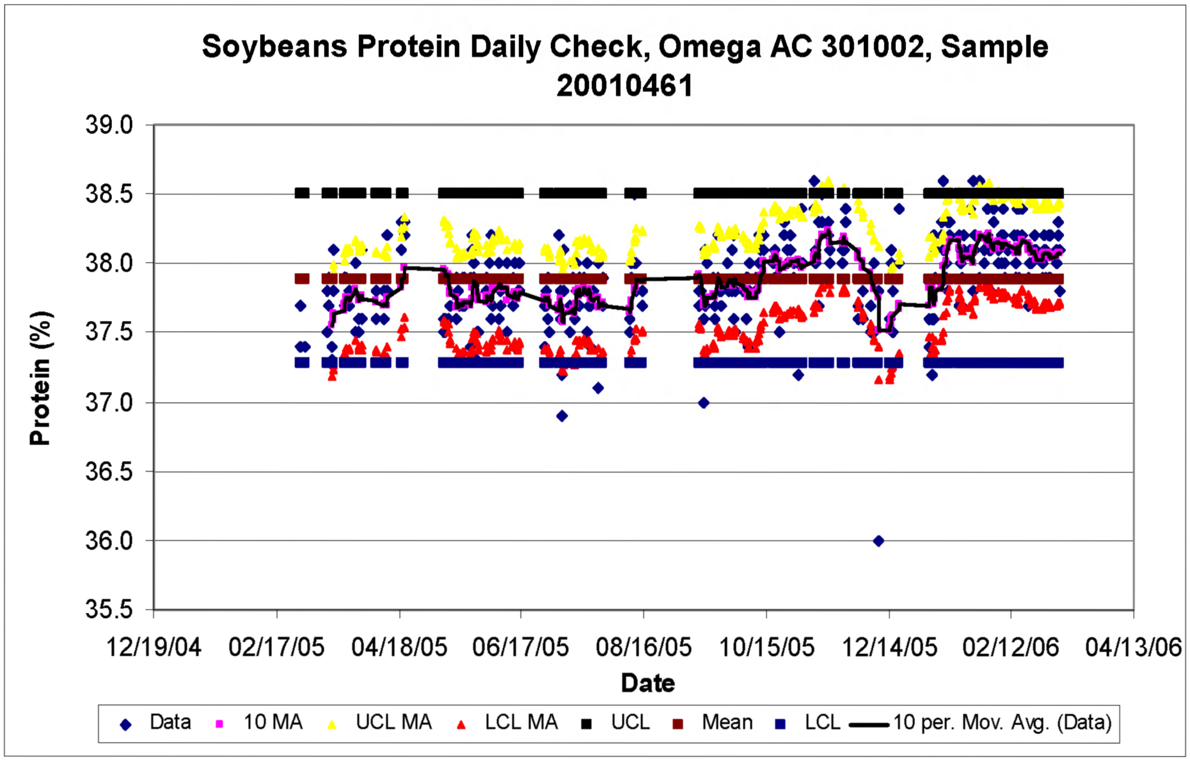


Figure 25. Soybeans Protein Daily Check, Sample 20010461, Omega AC 301002 Control Chart

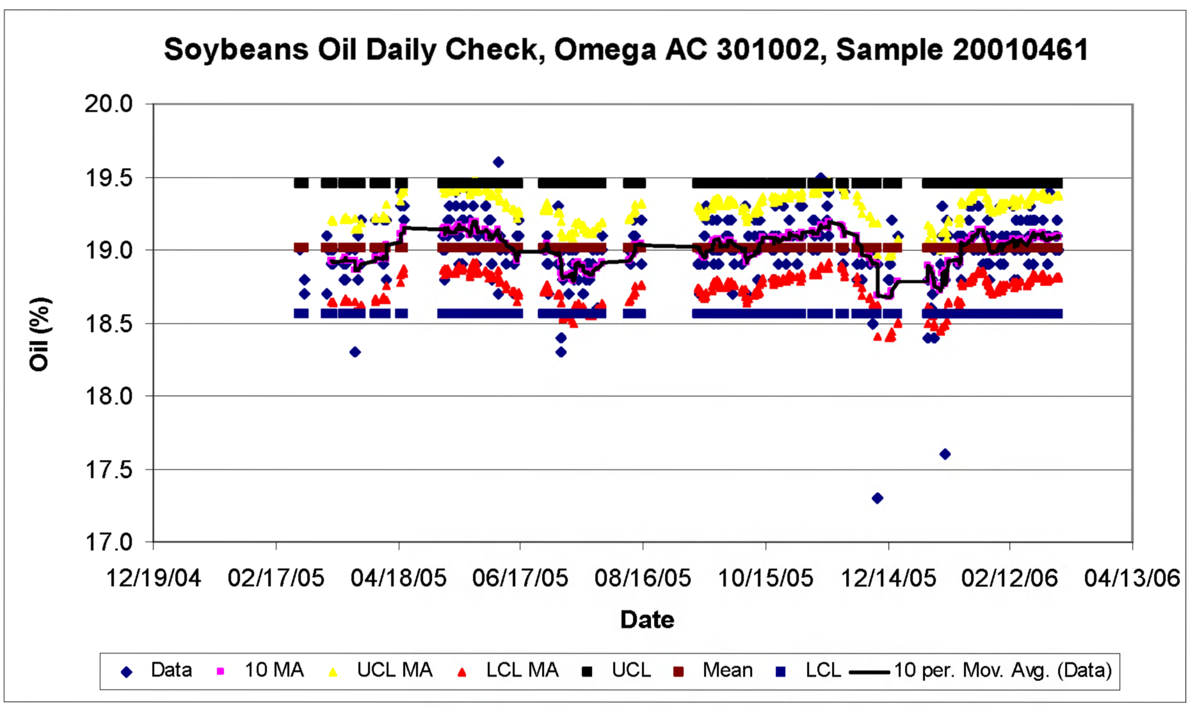


Figure 26. Soybeans Oil Daily Check, Sample 20010461, Omega AC 301002 Control Chart

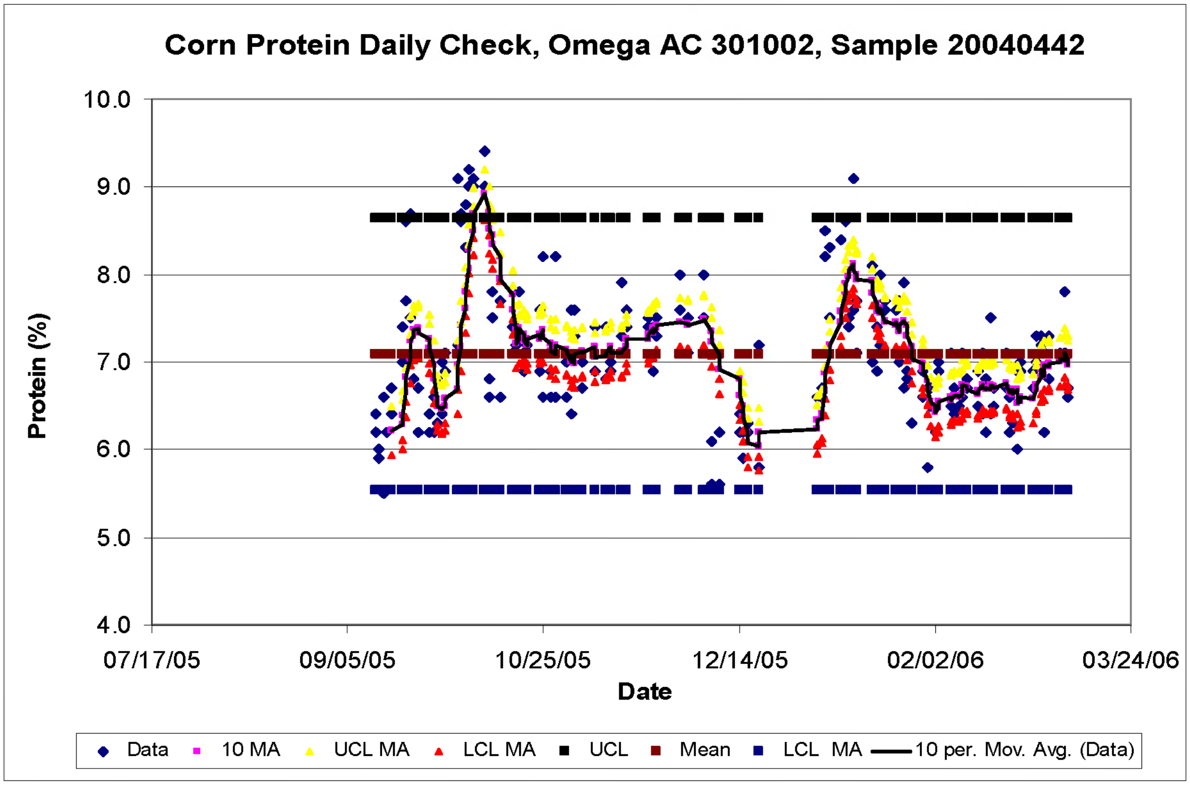


Figure 27. Corn Protein Daily Check, Sample 20040442, Omega AC 301002 Control Chart

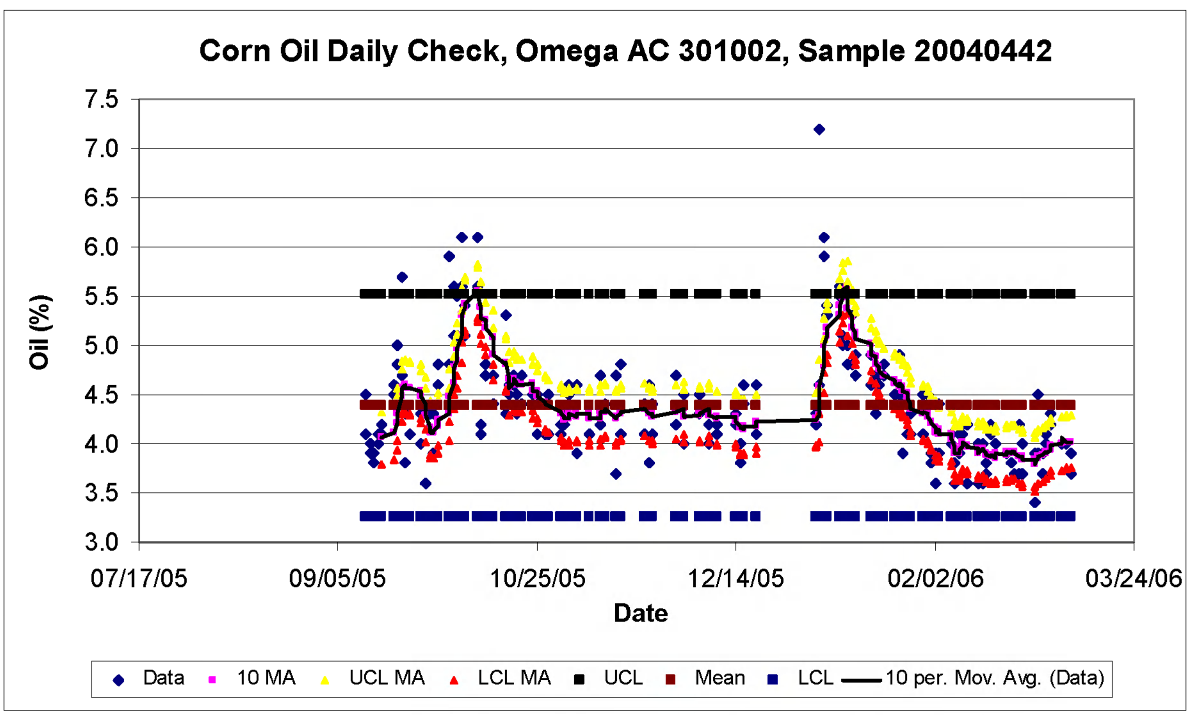


Figure 28. Corn Oil Daily Check, Sample 20040442, Omega AC 301002 Control Chart

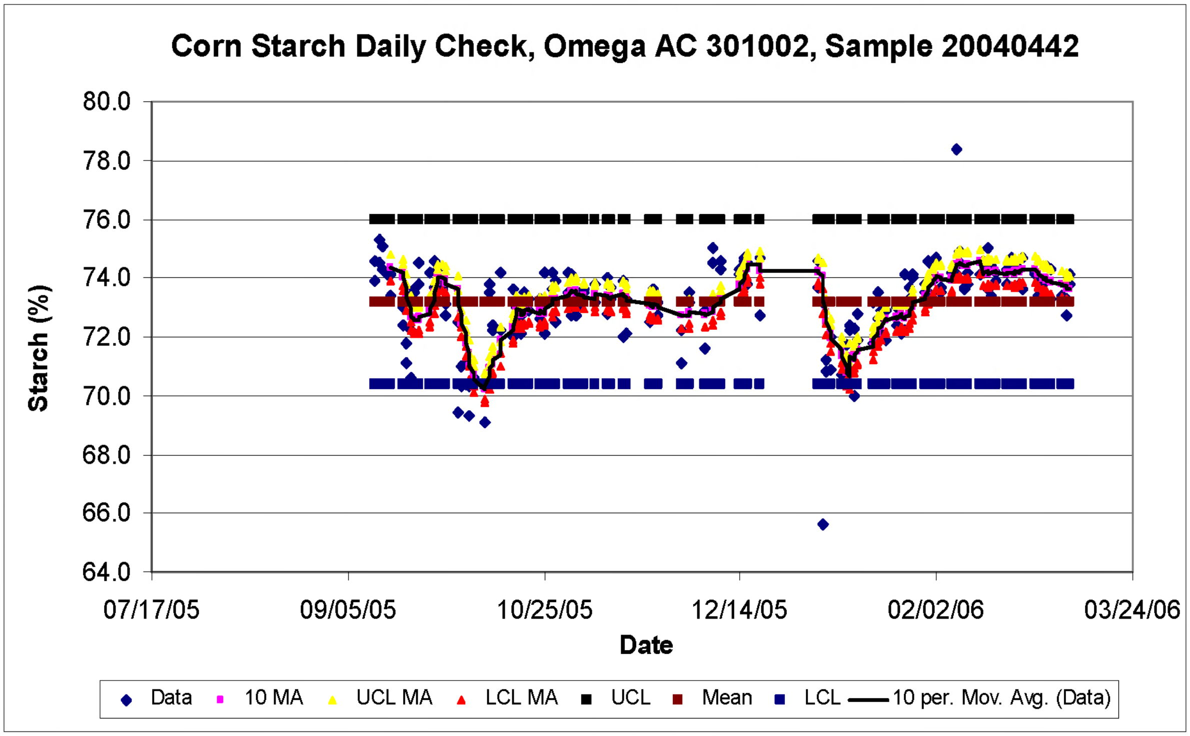


Figure 29. Corn Starch Daily Check, Sample 20040442, Omega AC 301002 Control Chart

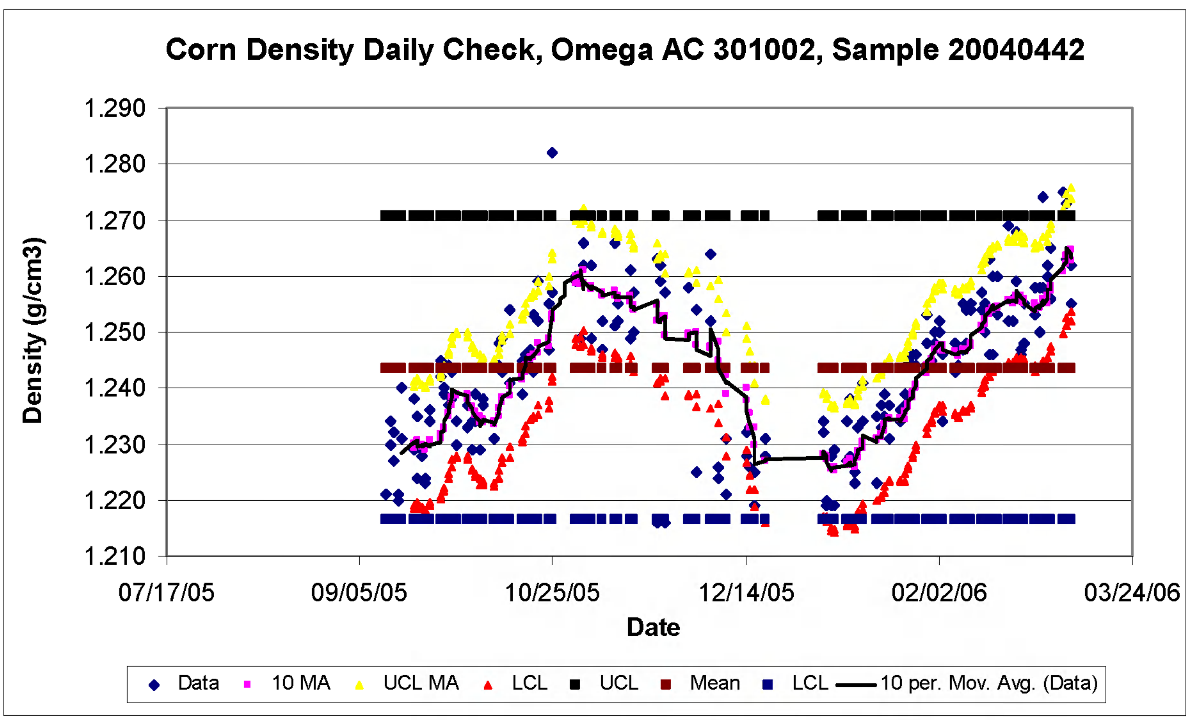


Figure 30. Corn Density Daily Check, Sample 20040442, Omega AC 301002 Control Chart

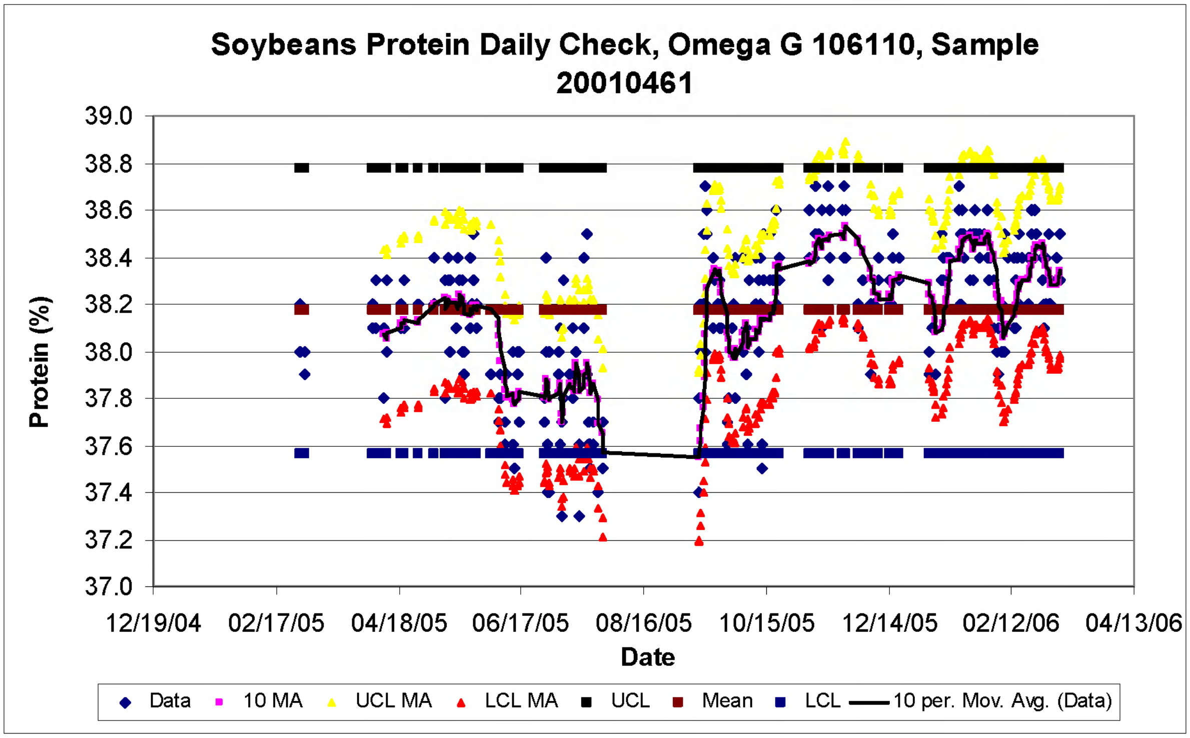


Figure 31. Soybeans Protein Daily Check, Sample 20010461, Omega G 106110 Control Chart

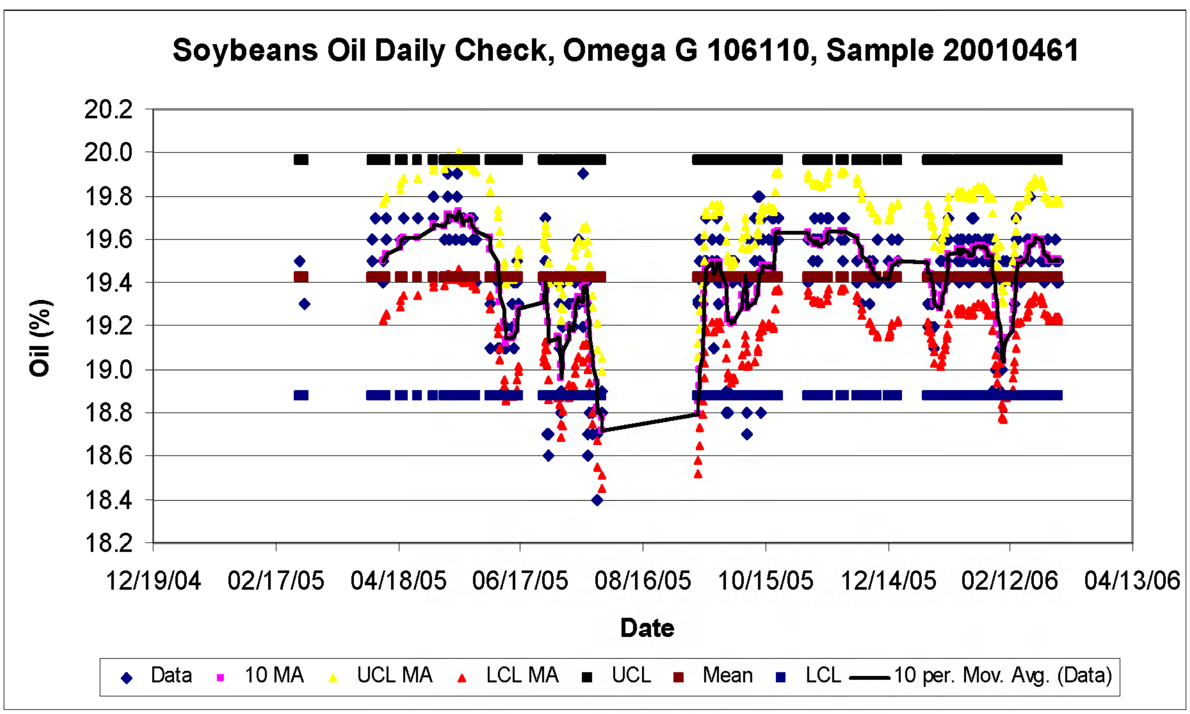


Figure 32. Soybeans Oil Daily Check, Sample 20010461, Omega G 106110 Control Chart

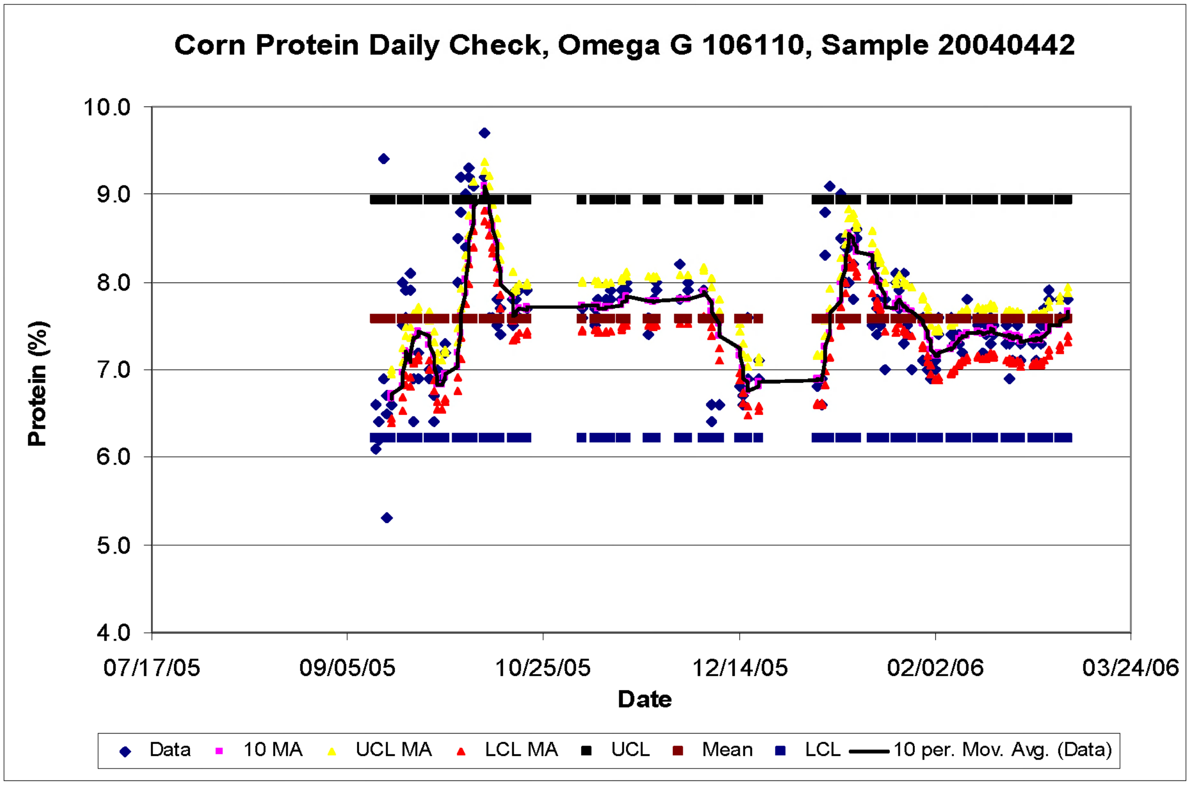


Figure 33. Corn Protein Daily Check, Sample 20040442, Omega G 106110 Control Chart

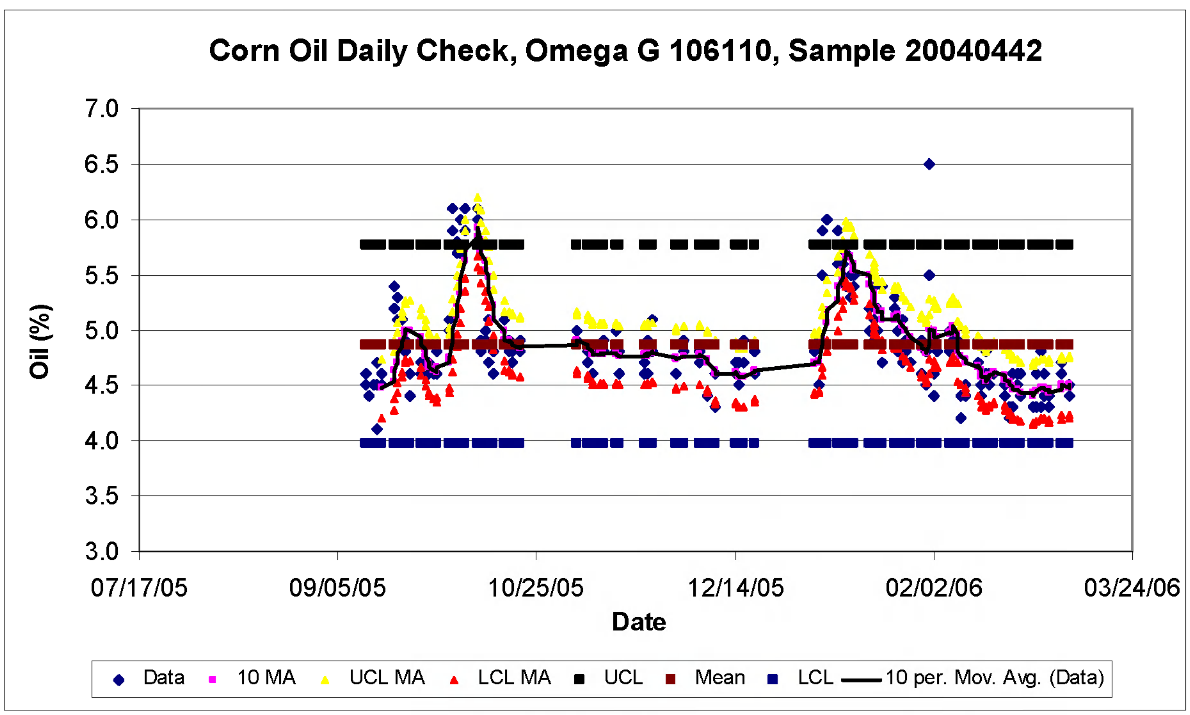


Figure 34. Corn Oil Daily Check, Sample 20040442, Omega G 106110 Control Chart

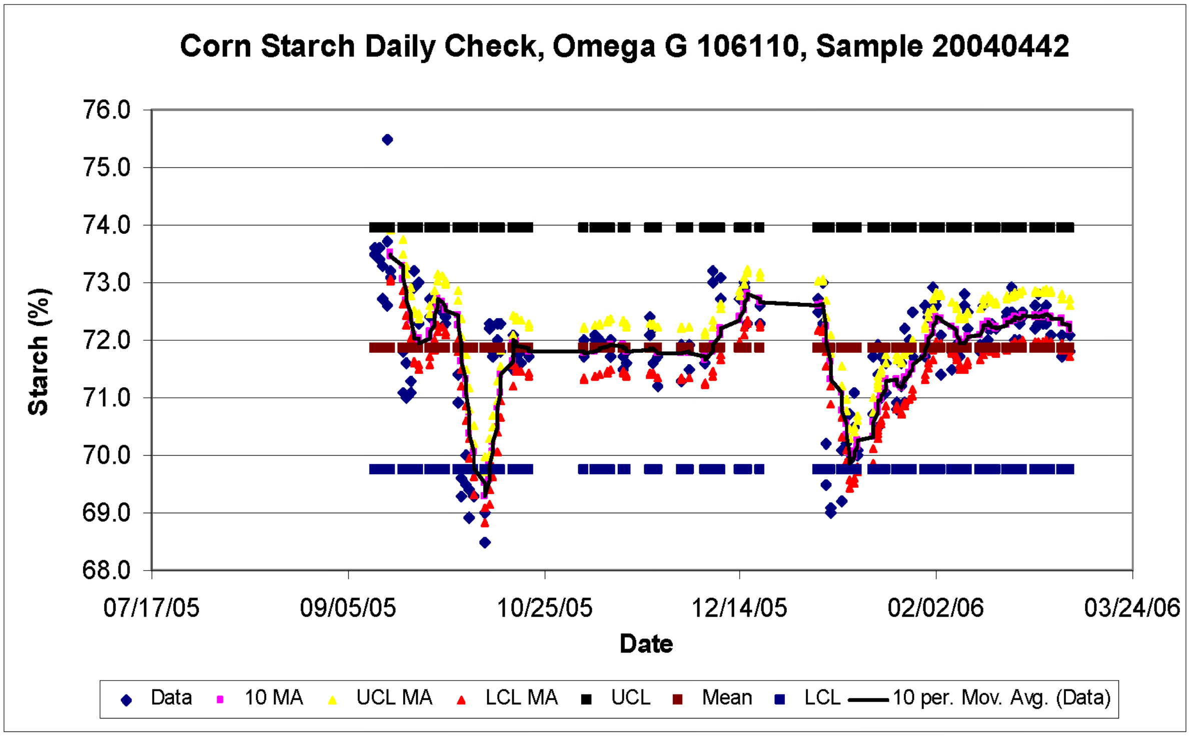


Figure 35. Corn Starch Daily Check, Sample 20040442, Omega G 106110 Control Chart

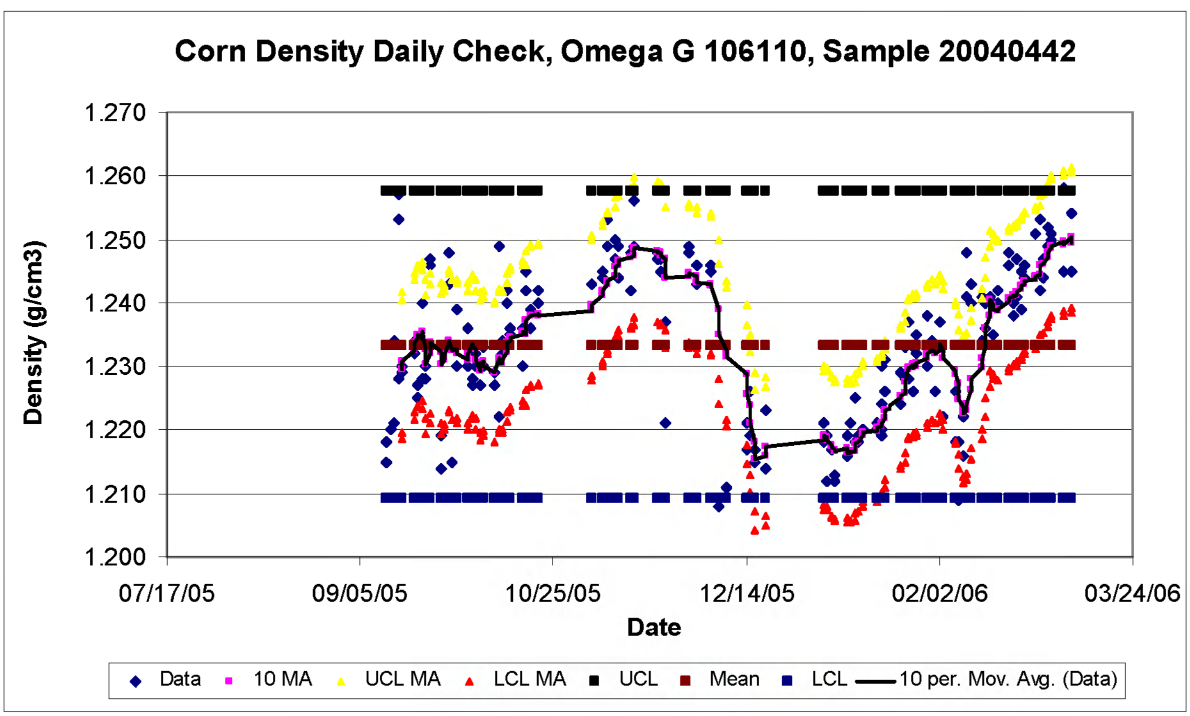


Figure 36. Corn Density Daily Check, Sample 20040442, Omega G 106110 Control Chart

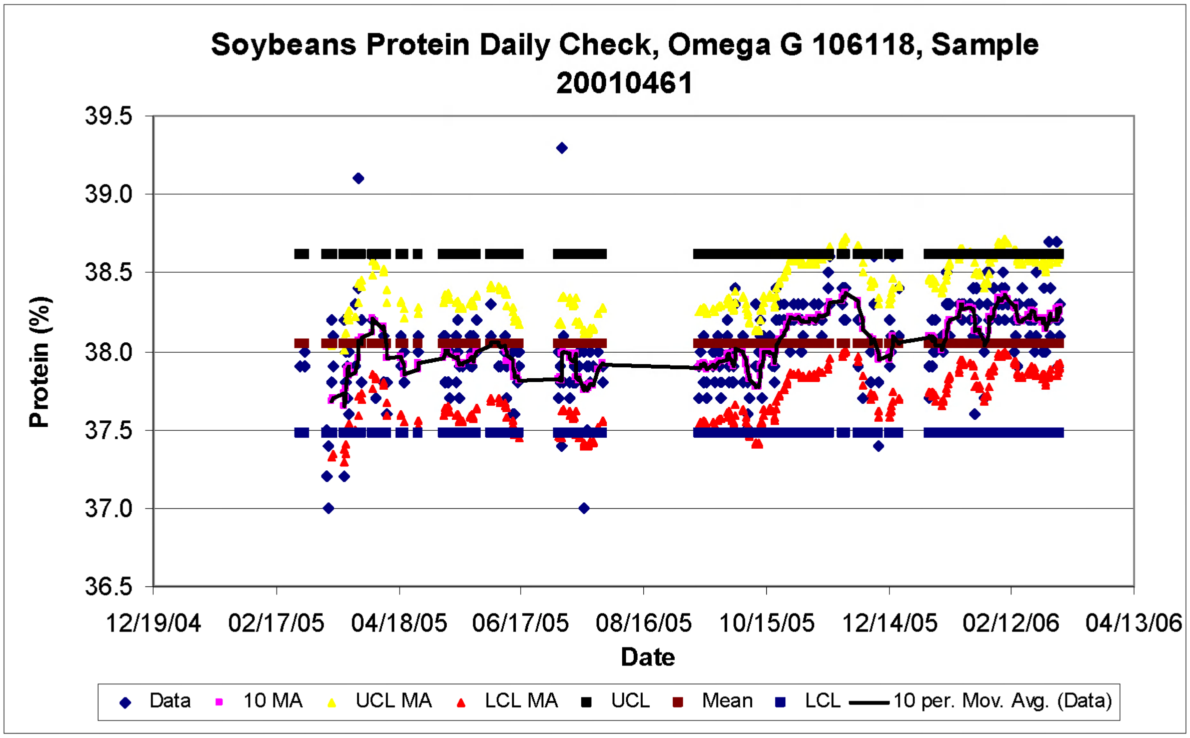


Figure 37. Soybeans Protein Daily Check, Sample 20010461, Omega G 106118 Control Chart

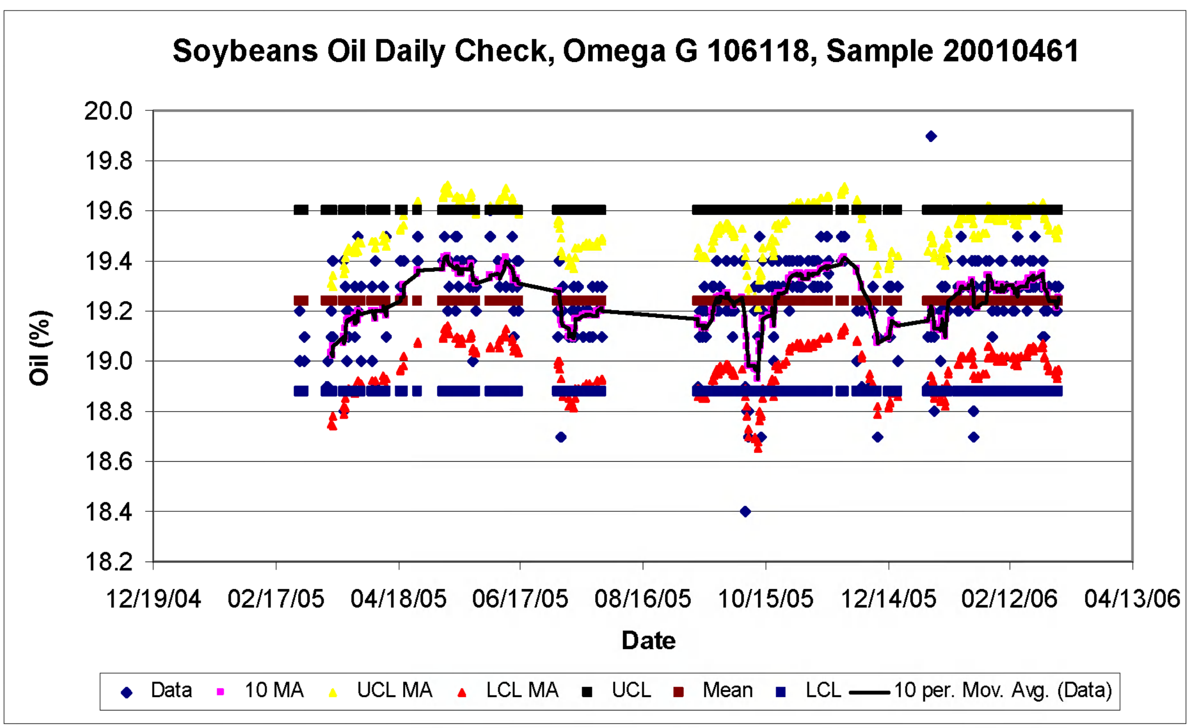


Figure 38. Soybeans Oil Daily Check, Sample 20010461, Omega G 106118 Control Chart

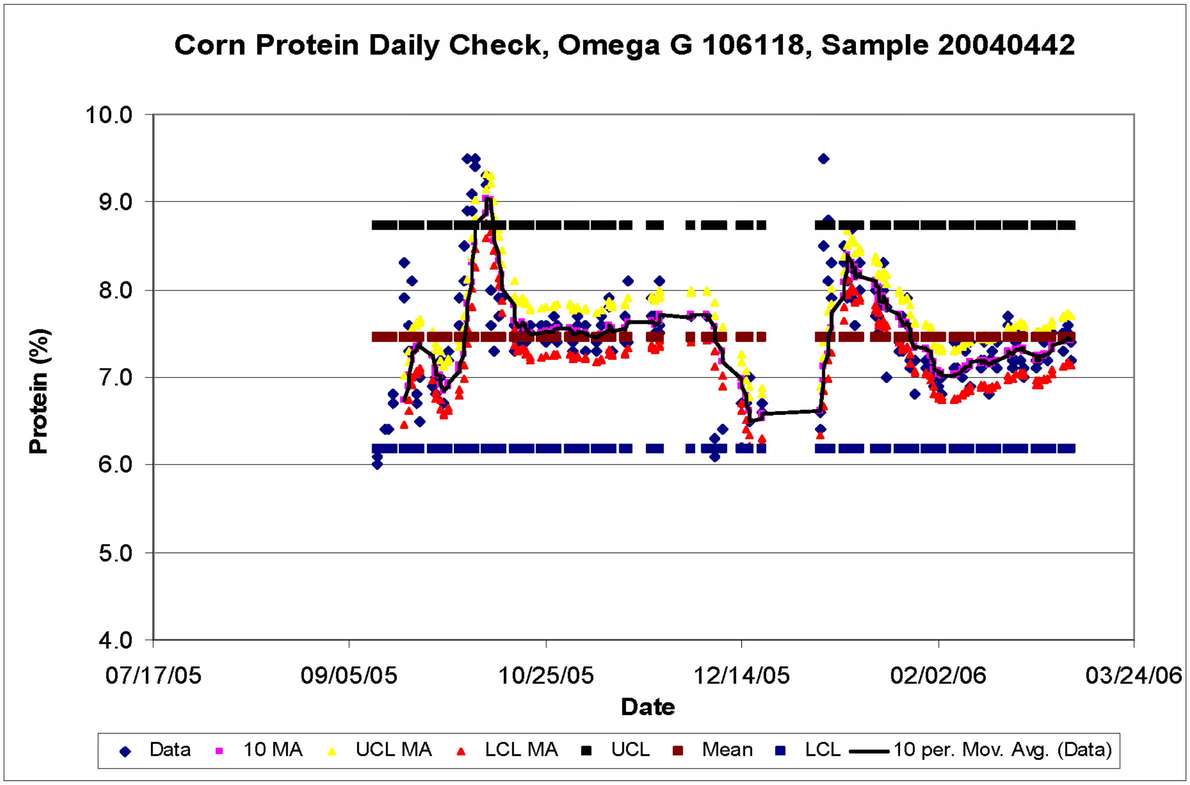


Figure 39. Corn Protein Daily Check, Sample 20040442, Omega G 106118 Control Chart

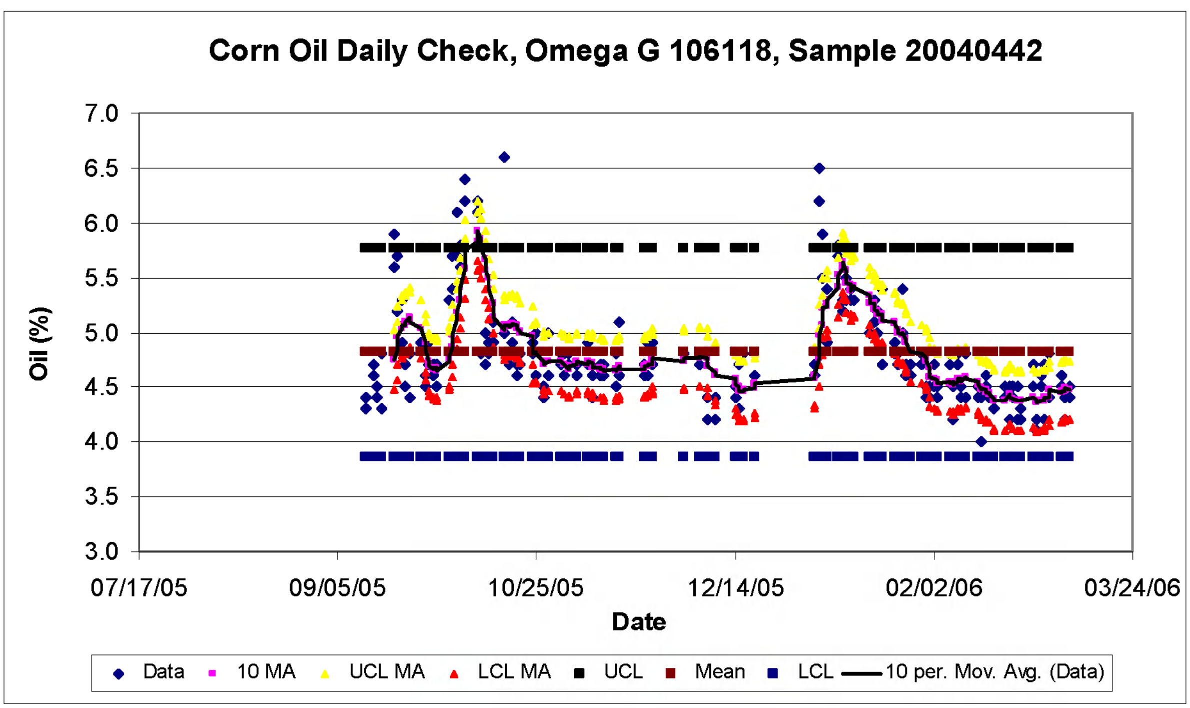


Figure 40. Corn Oil Daily Check, Sample 20040442, Omega G 106118 Control Chart

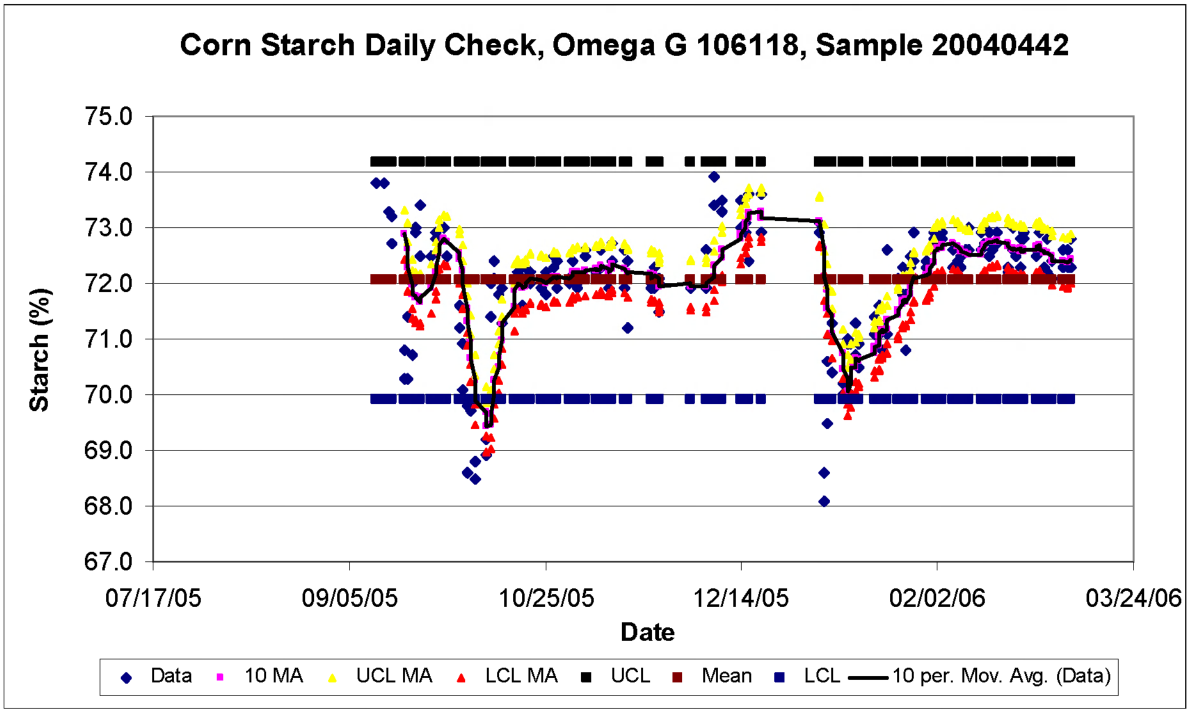


Figure 41. Corn Starch Daily Check, Sample 20040442, Omega G 106118 Control Chart

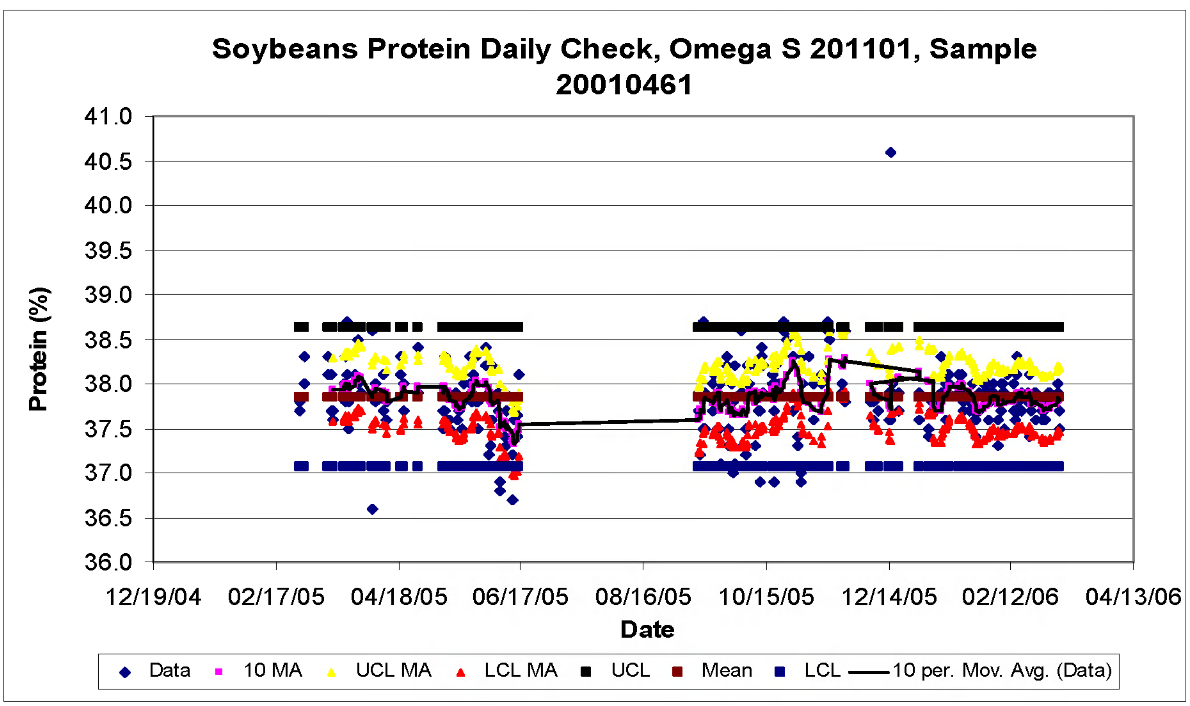


Figure 42. Soybeans Protein Daily Check, Sample 20010461, Omega S 201101 Control Chart

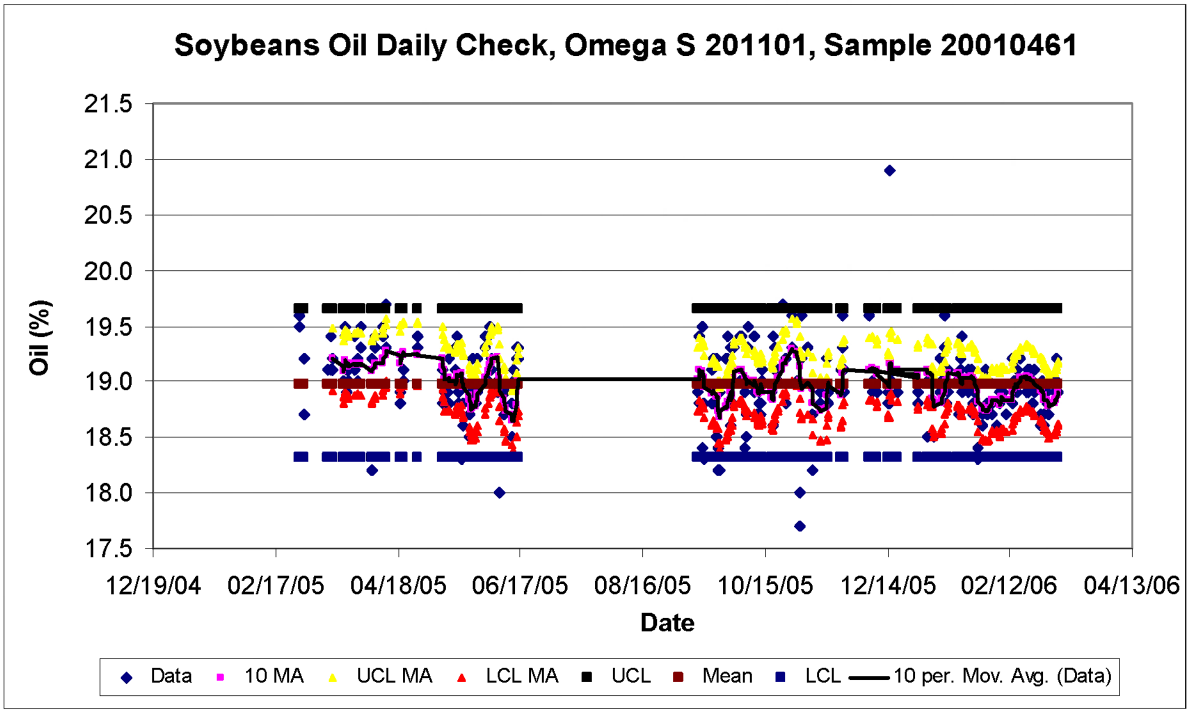


Figure 43. Soybeans Oil Daily Check, Sample 20010461, Omega S 201101 Control Chart

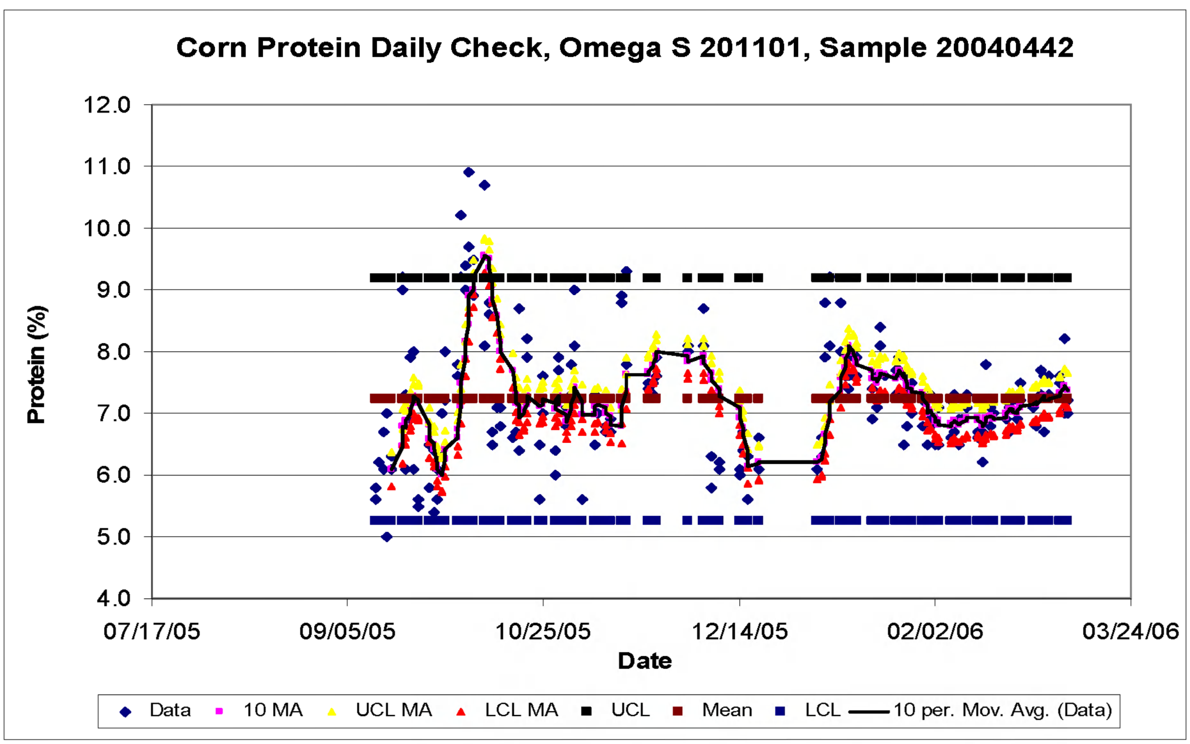


Figure 44. Corn Protein Daily Check, Sample 20040442, Omega S 201101 Control Chart

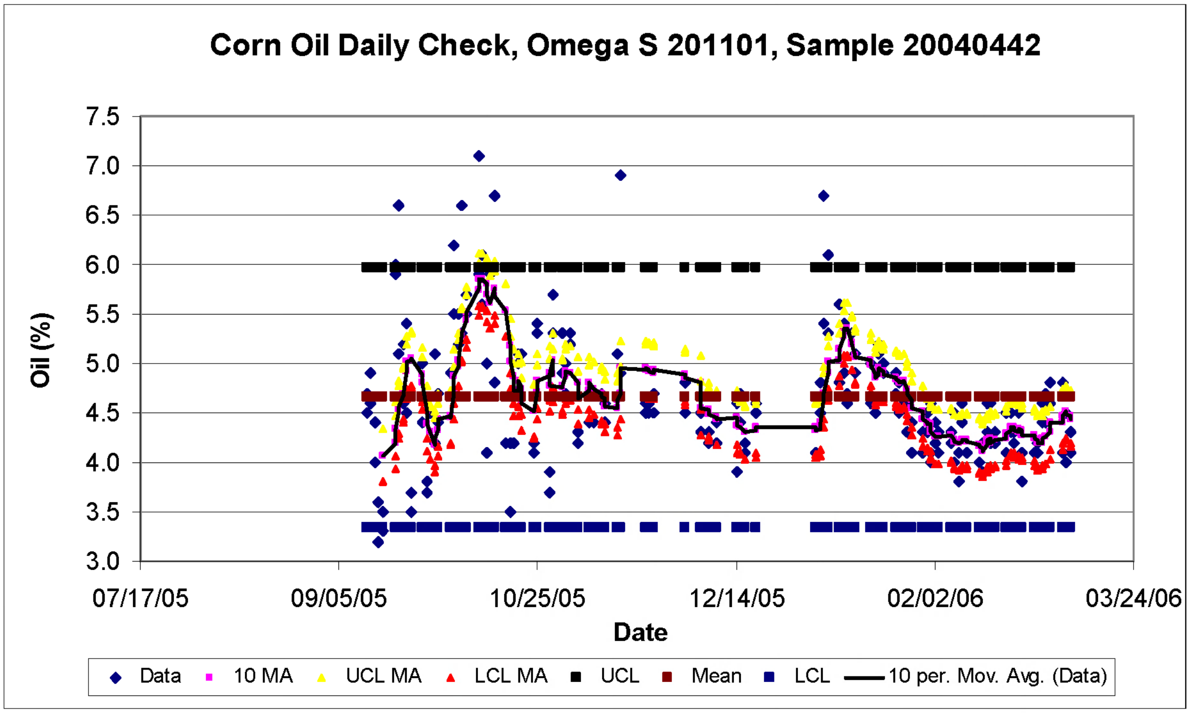


Figure 45. Corn Oil Daily Check, Sample 20040442, Omega S 201101 Control Chart

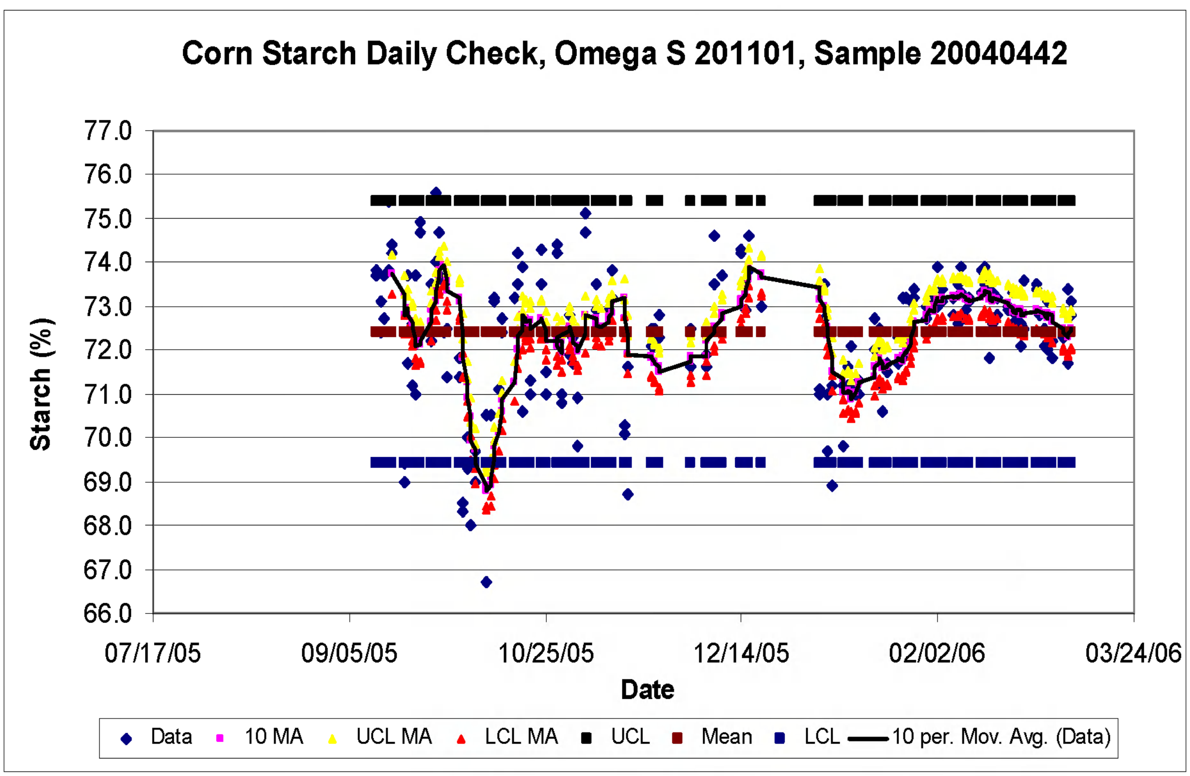


Figure 46. Corn Starch Daily Check, Sample 20040442, Omega S 201101 Control Chart

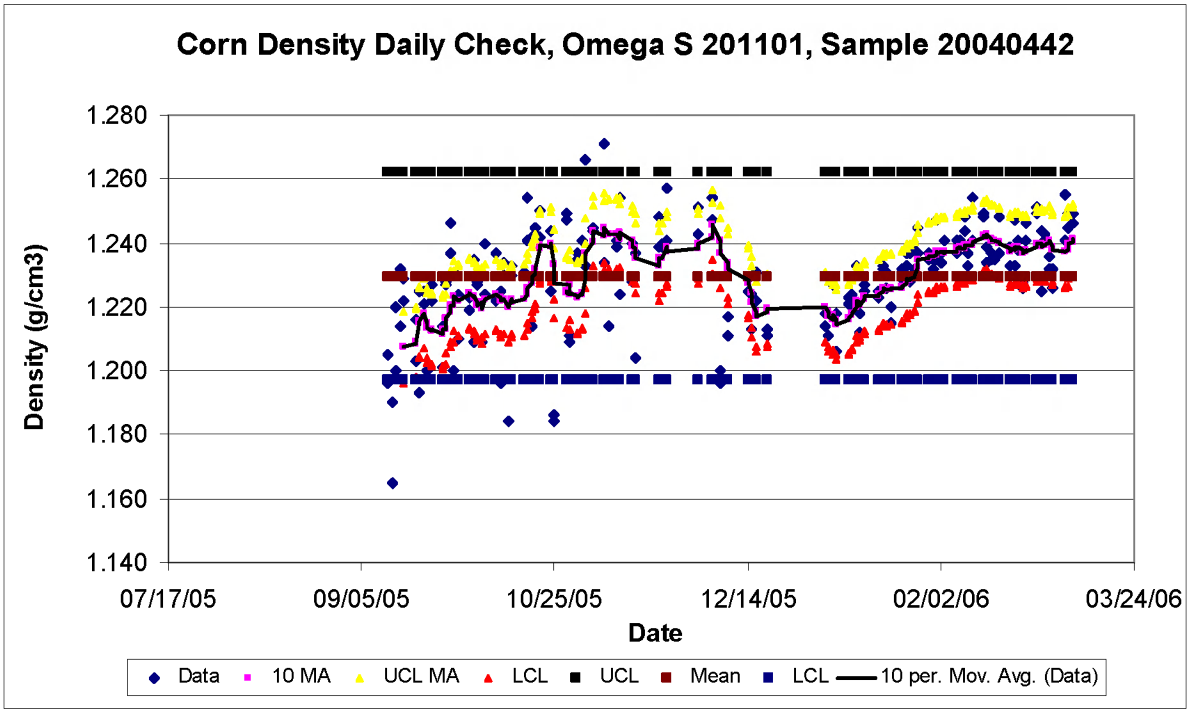


Figure 47. Corn Density Daily Check, Sample 20040442, Omega S 201101 Control Chart

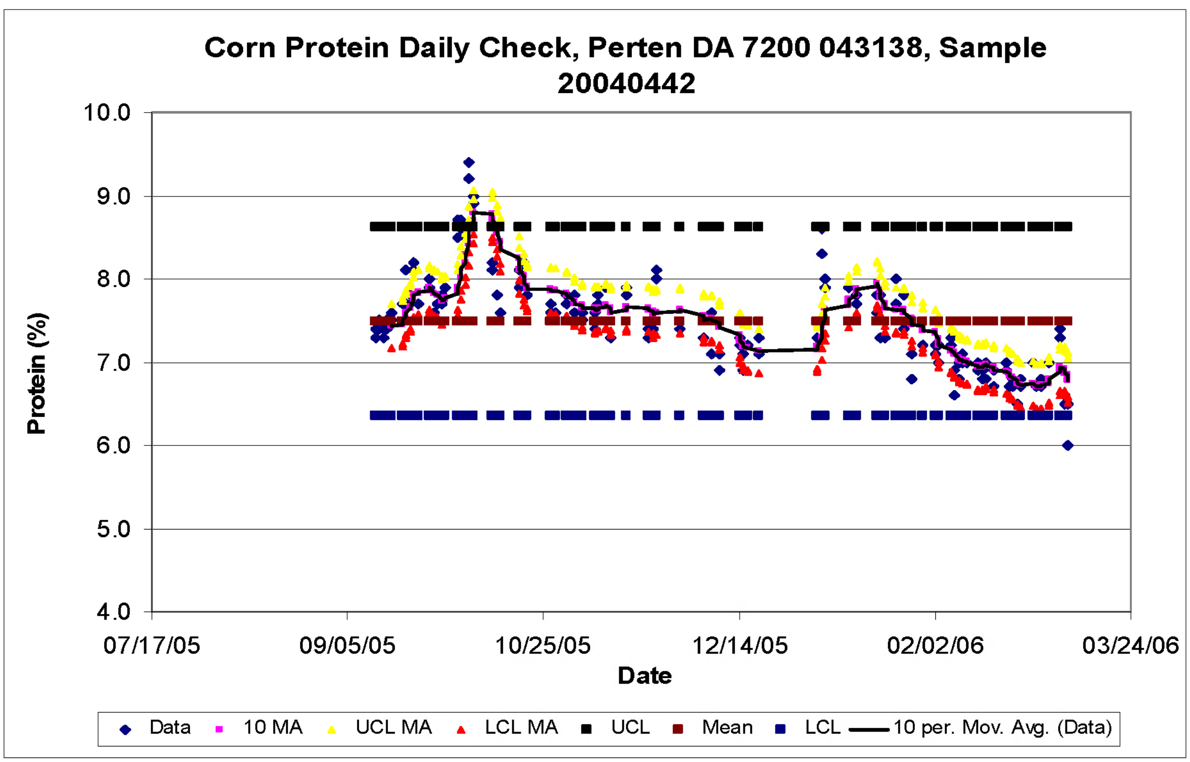


Figure 48. Corn Protein Daily Check, Sample 20040442, Perten DA 7200 043138 Control Chart

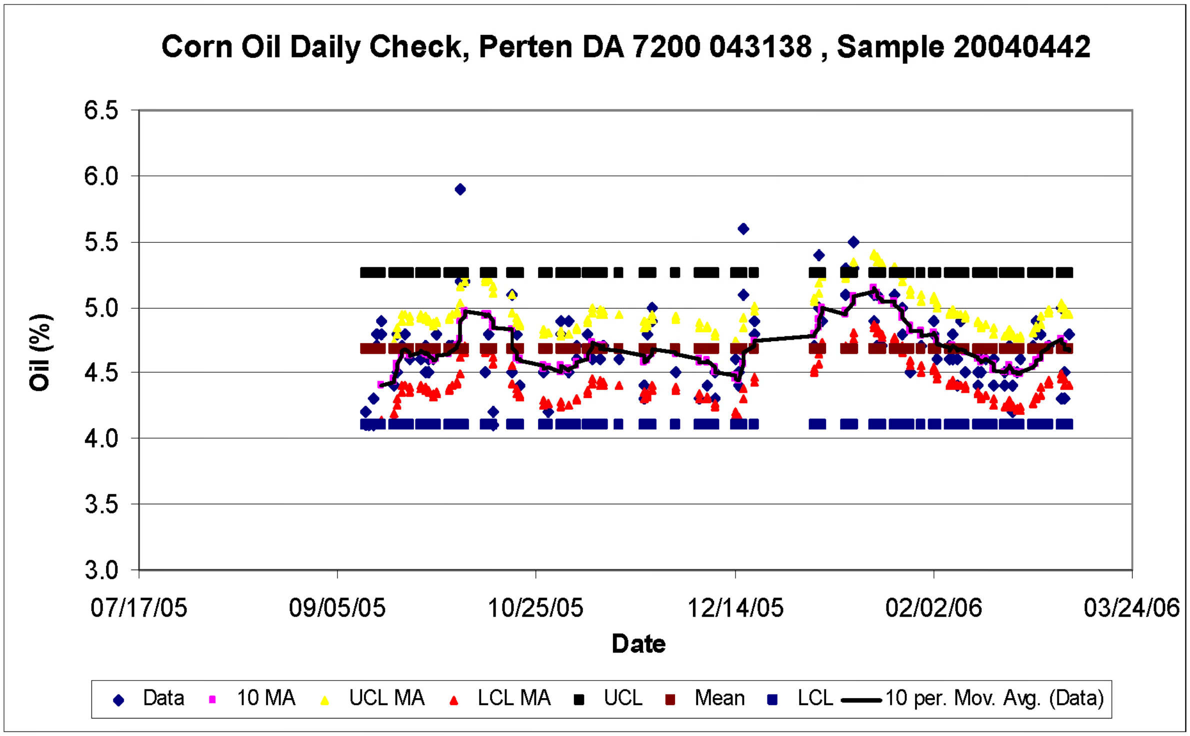


Figure 49. Corn Oil Daily Check, Sample 20040442, Perten DA 7200 043138 Control Chart

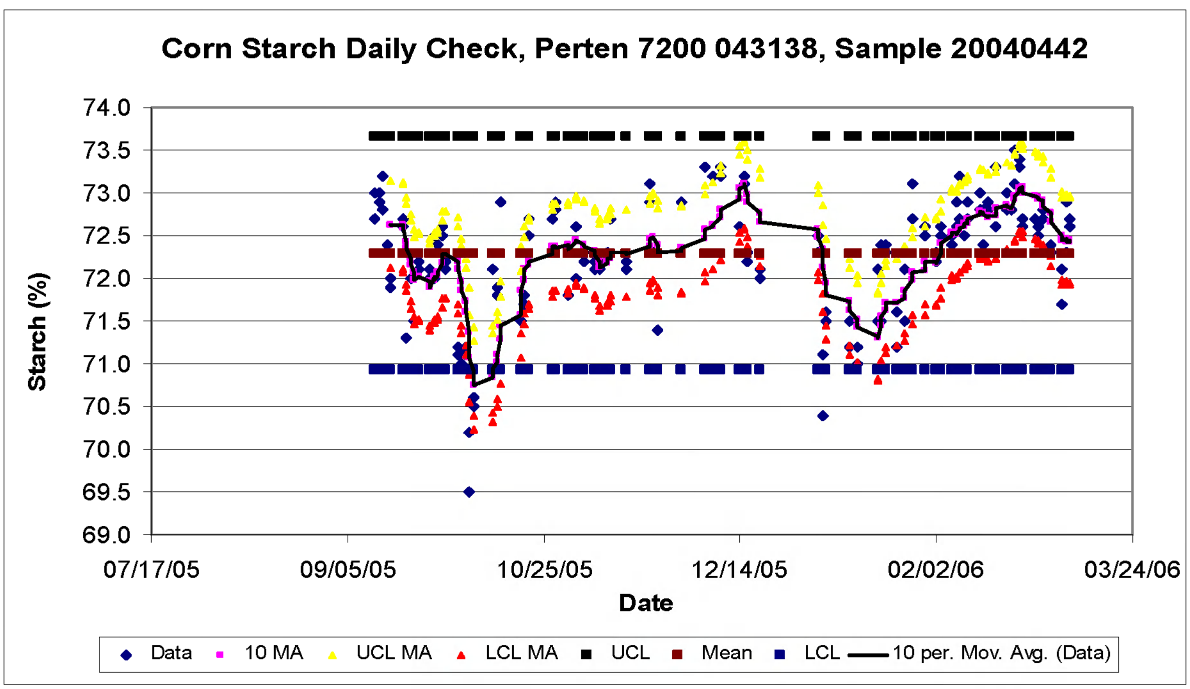


Figure 50. Corn Starch Daily Check, Sample 20040442, Perten DA 7200 043138 Control Chart

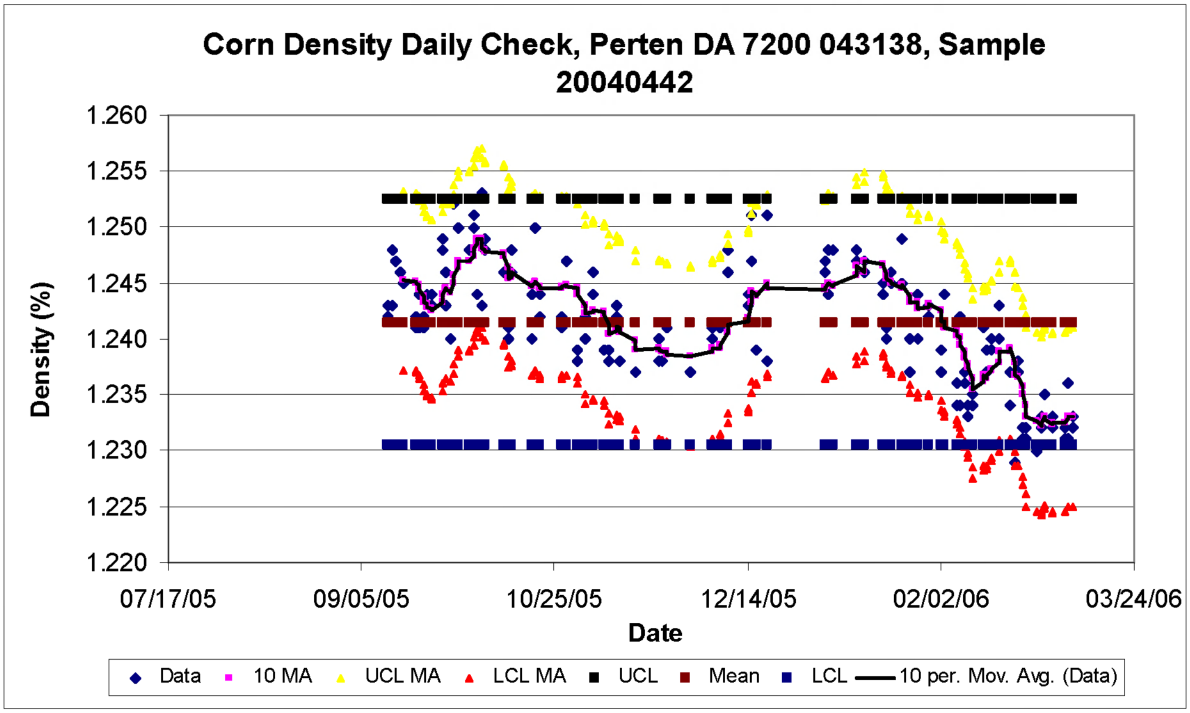


Figure 51. Corn Density Daily Check, Sample 20040442, Perten DA 7200 043138 Control Chart

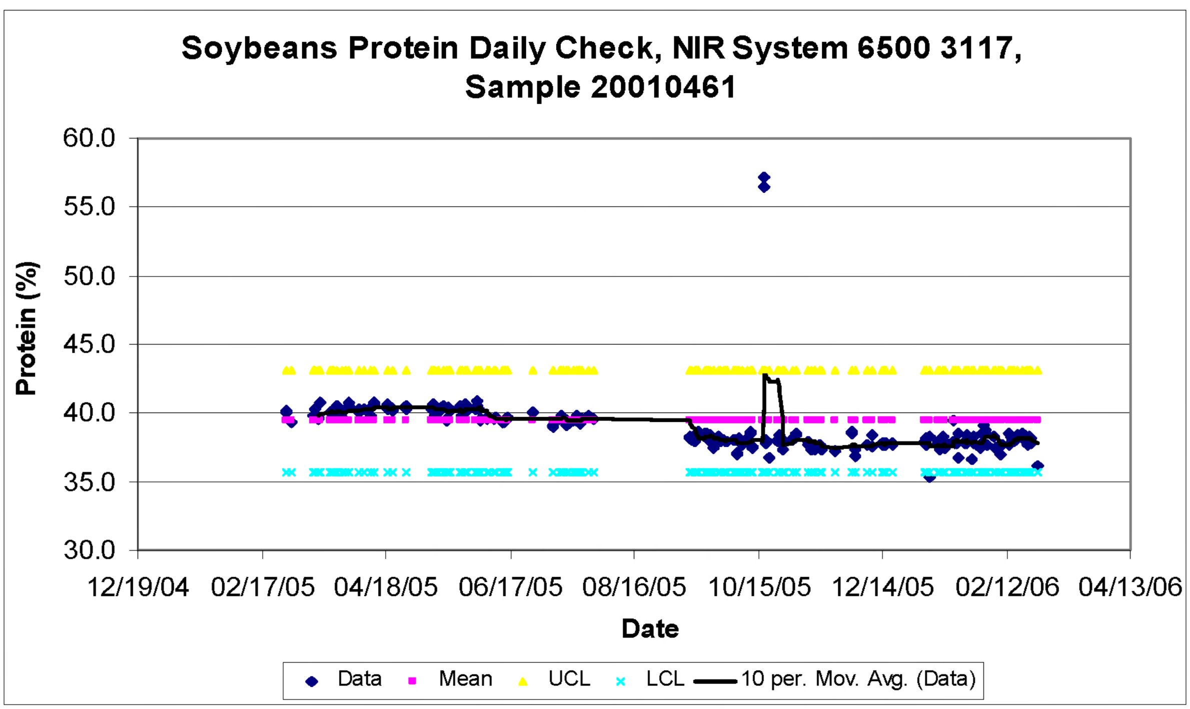


Figure 52. Soybeans Protein Daily Check, Sample 20010461, NIR System 6500 3117 Control Chart

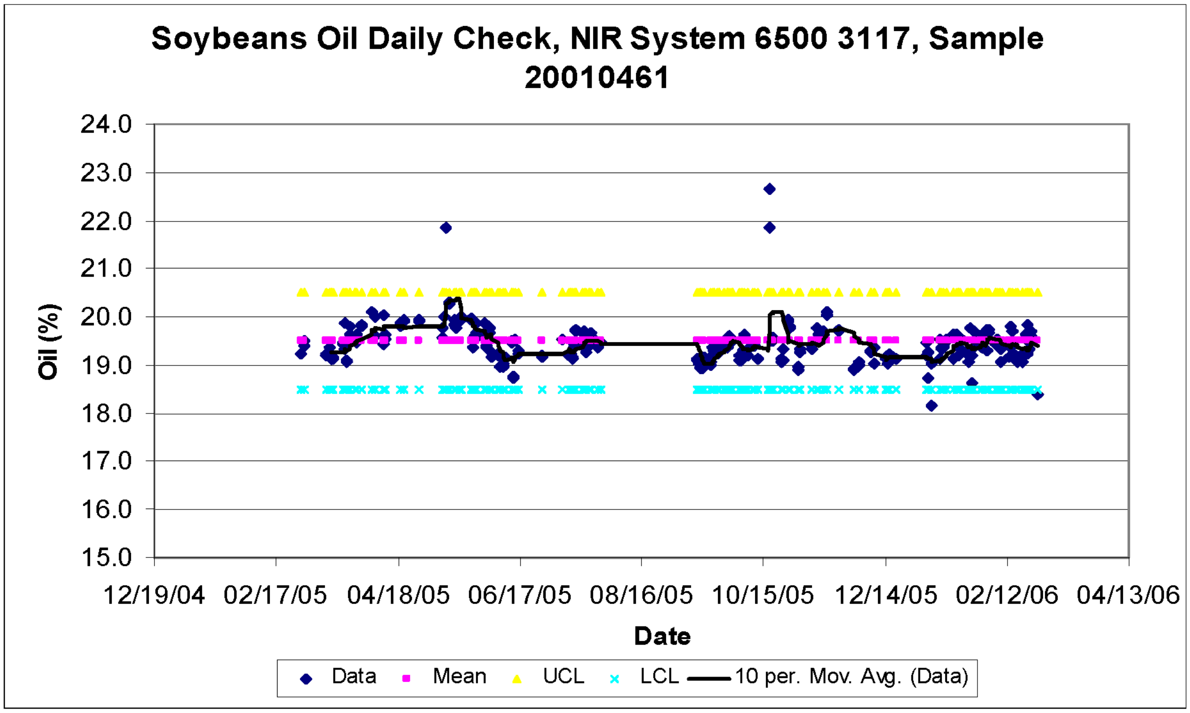


Figure 53. Soybeans Oil Daily Check, Sample 20010461, NIR System 6500 3117 Control Chart

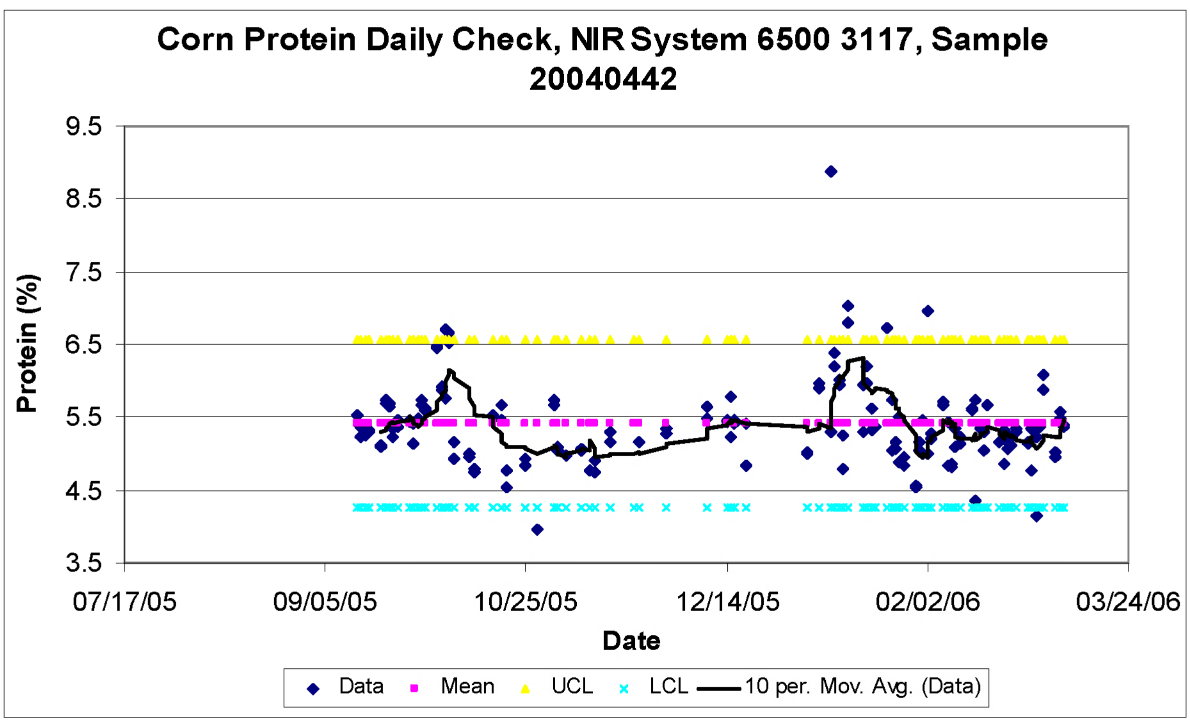


Figure 54. Corn Protein Daily Check, Sample 20040442, NIR System 6500 3117 Control Chart

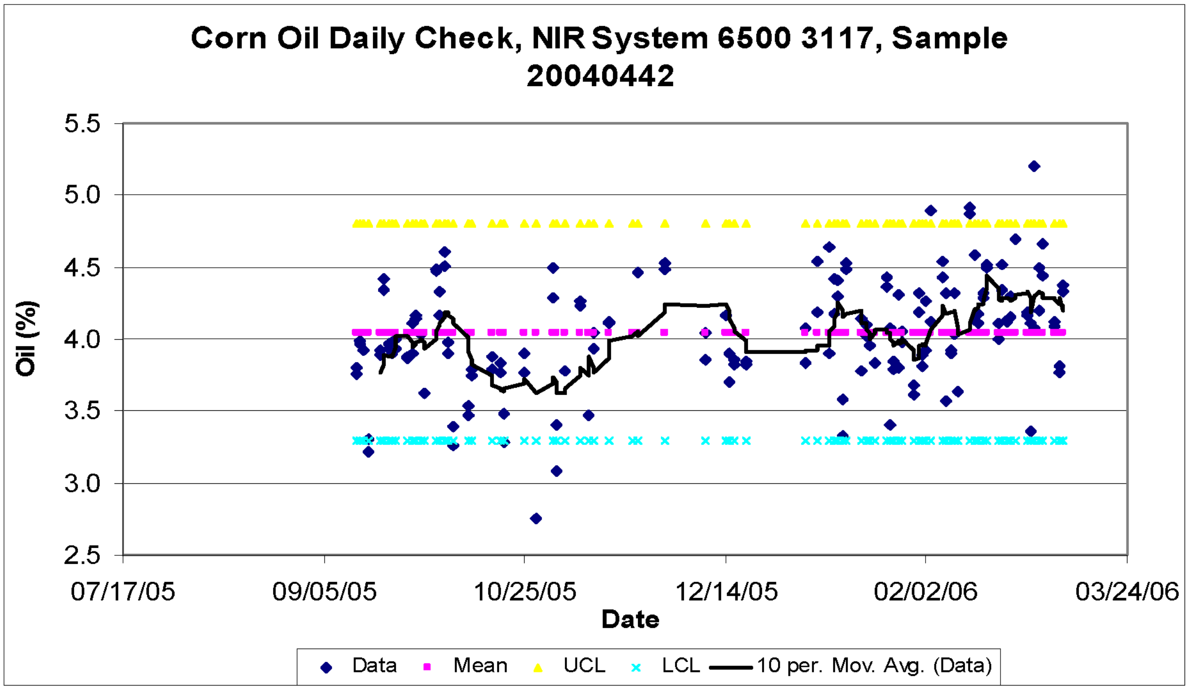


Figure 55. Corn Oil Daily Check, Sample 20040442, NIR System 6500 3117 Control Chart

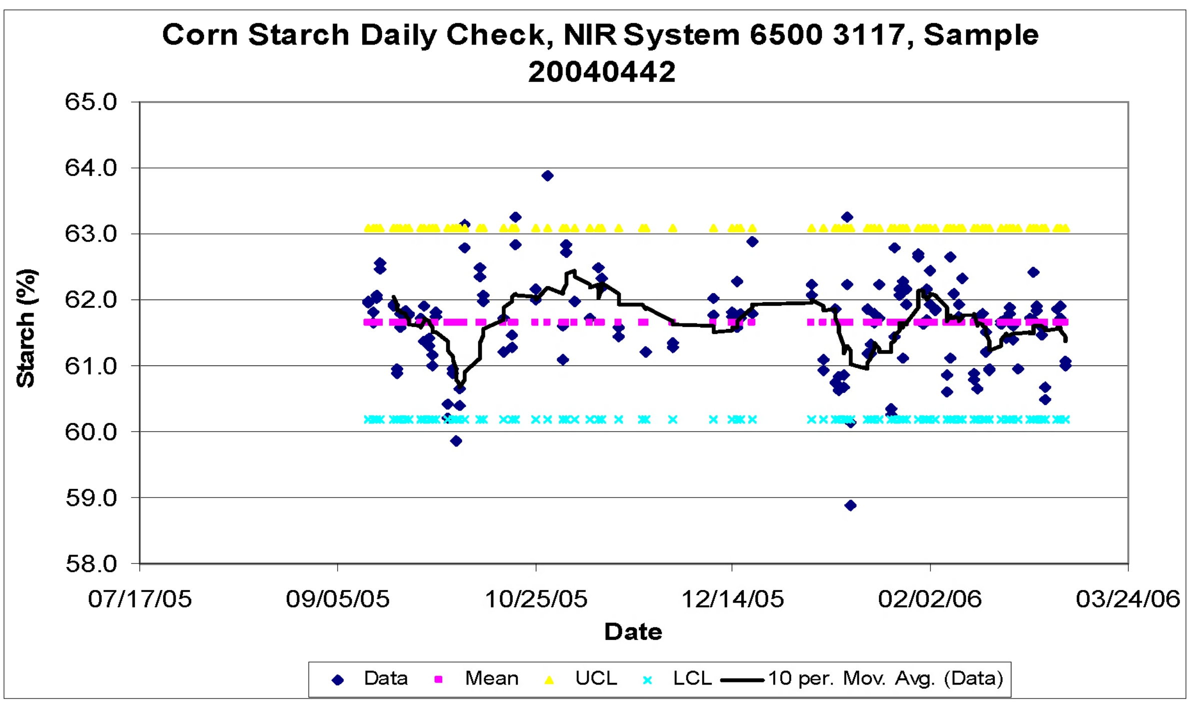


Figure 56. Corn Starch Daily Check, Sample 20040442, NIR System 6500 3117 Control Chart

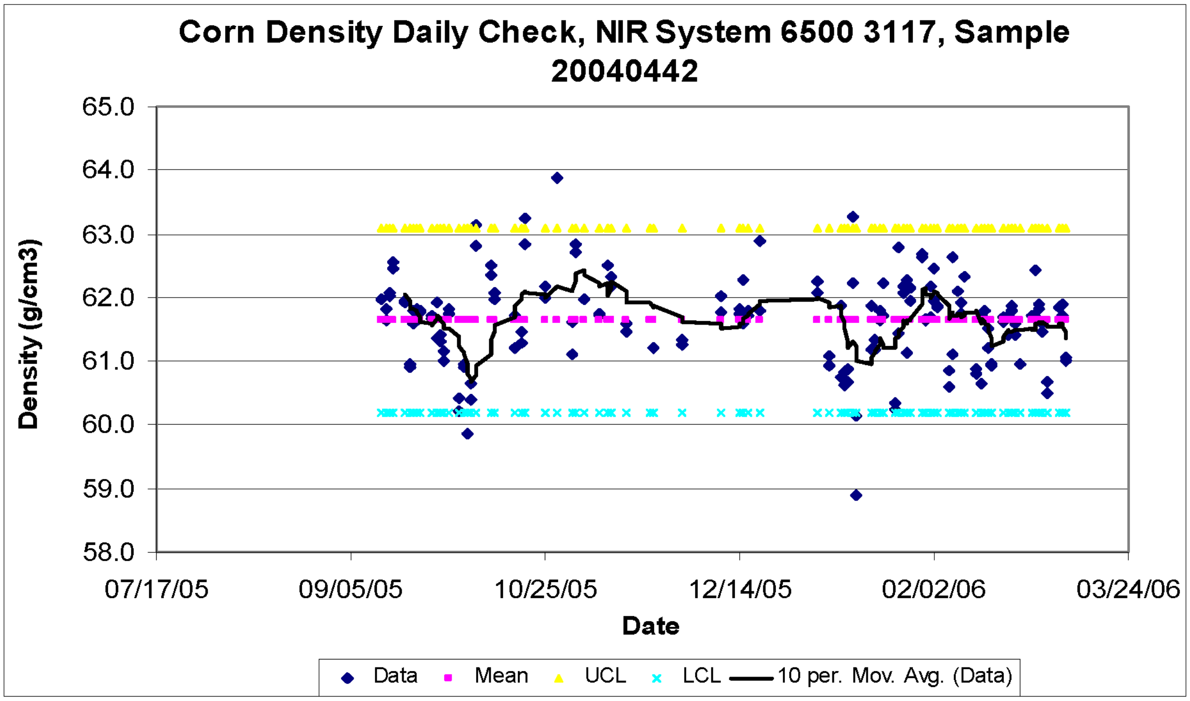


Figure 57. Corn Density Daily Check, Sample 20040442, NIR System 6500 3117 Control Chart

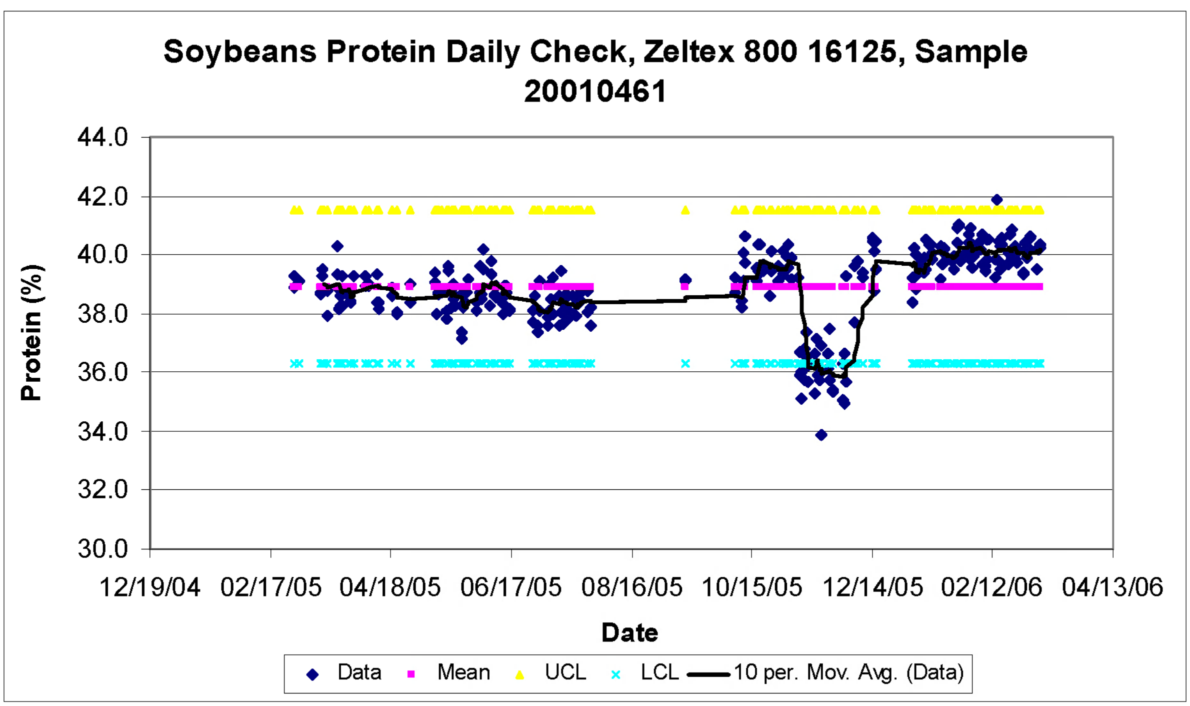


Figure 58. Soybeans Protein Daily Check, Sample 20010461, Zeltex 800 16125 Control Chart

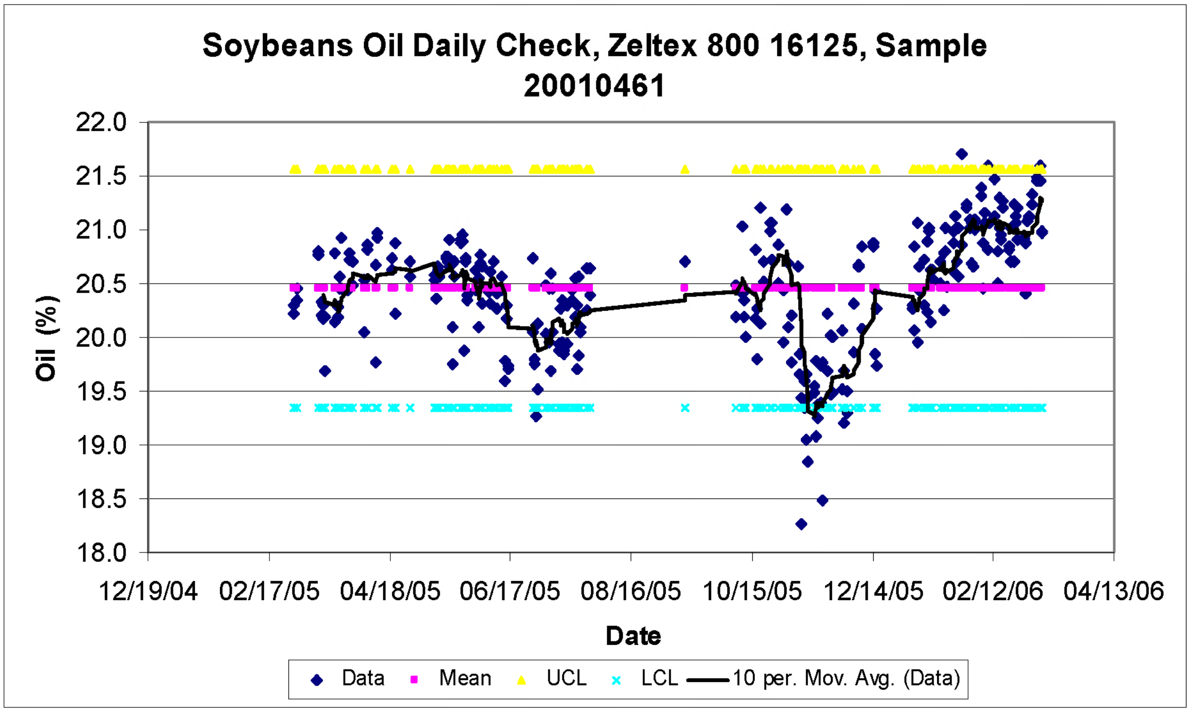


Figure 59. Soybeans Oil Daily Check, Sample 20010461, Zeltex 800 16125 Control Chart

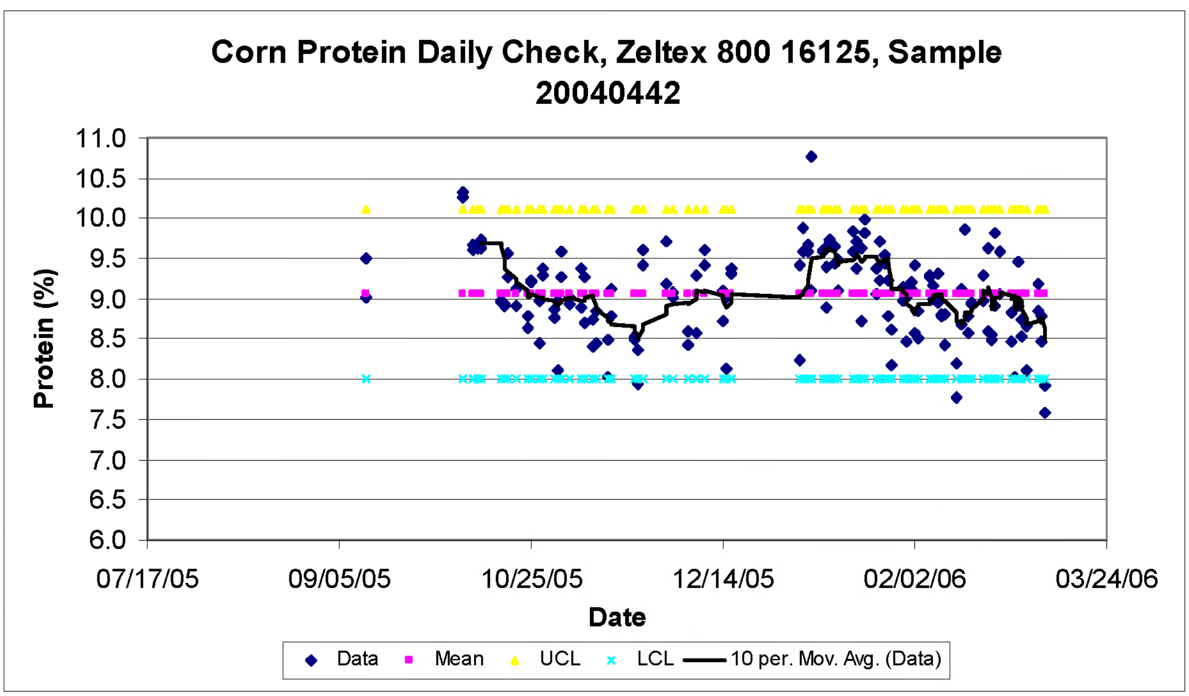


Figure 60. Corn Protein Daily Check, Sample 20040442, Zeltex 800 16125 Control Chart

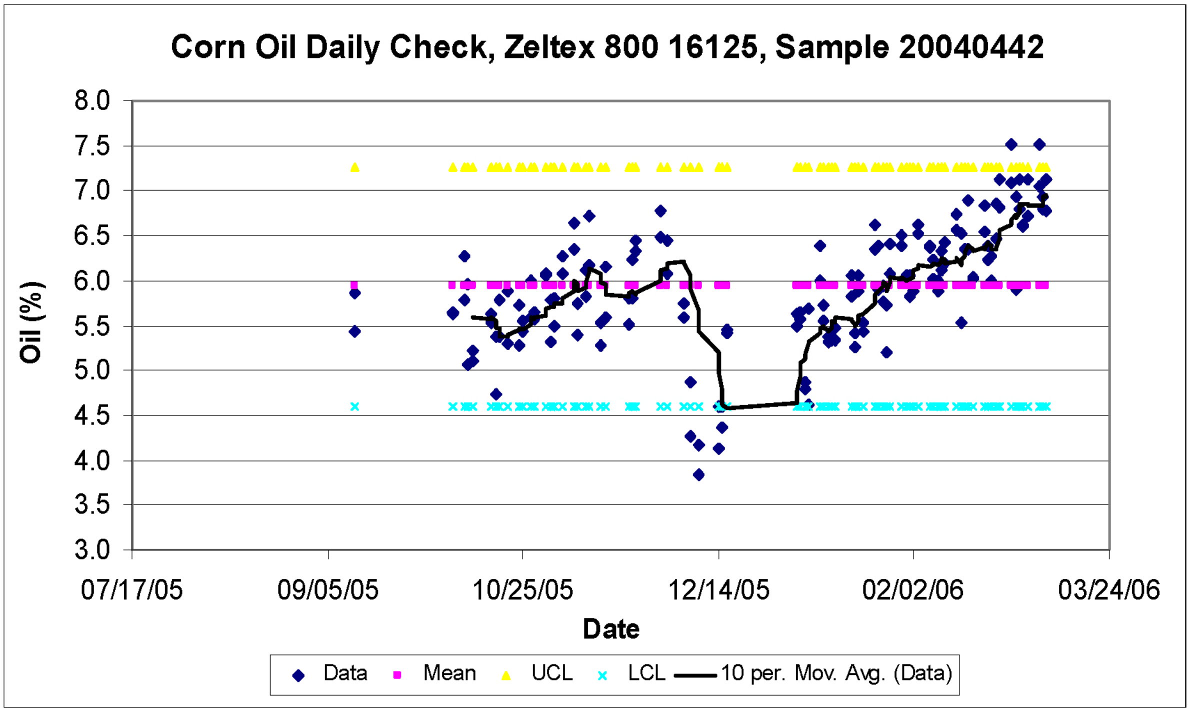


Figure 61. Corn Oil Daily Check, Sample 20040442, Zeltex 800 16125 Control Chart

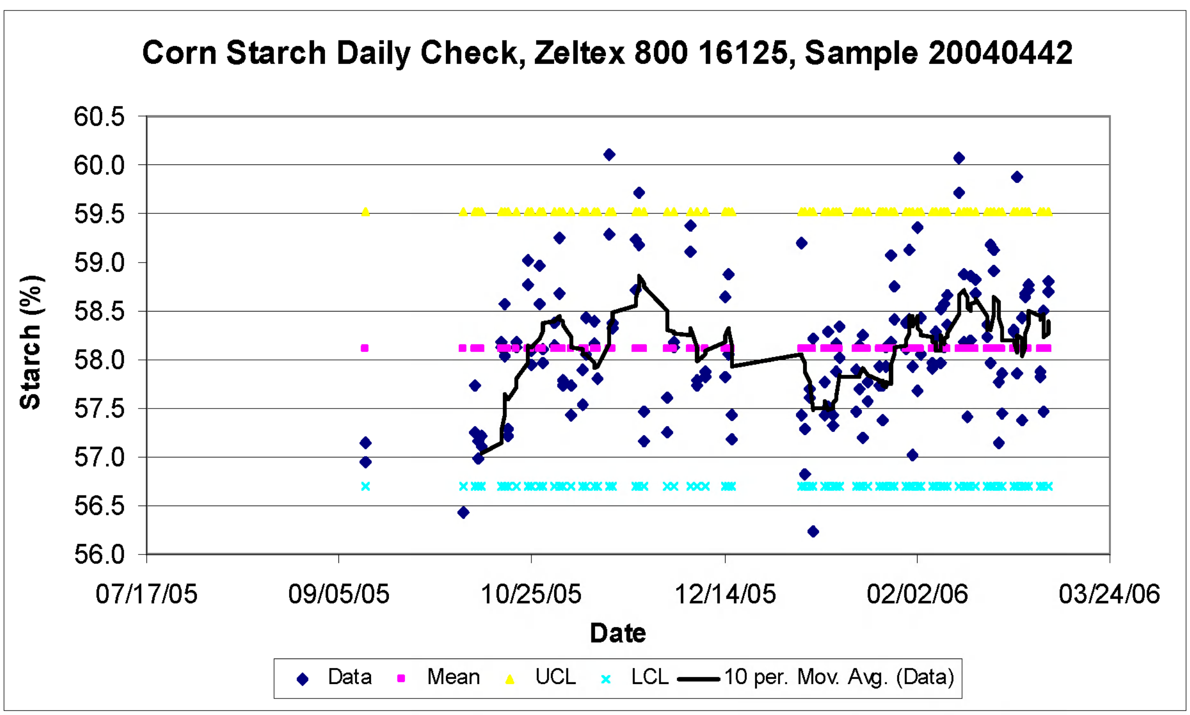


Figure 62. Corn Starch Daily Check, Sample 20040442, Zeltex 800 16125 Control Chart

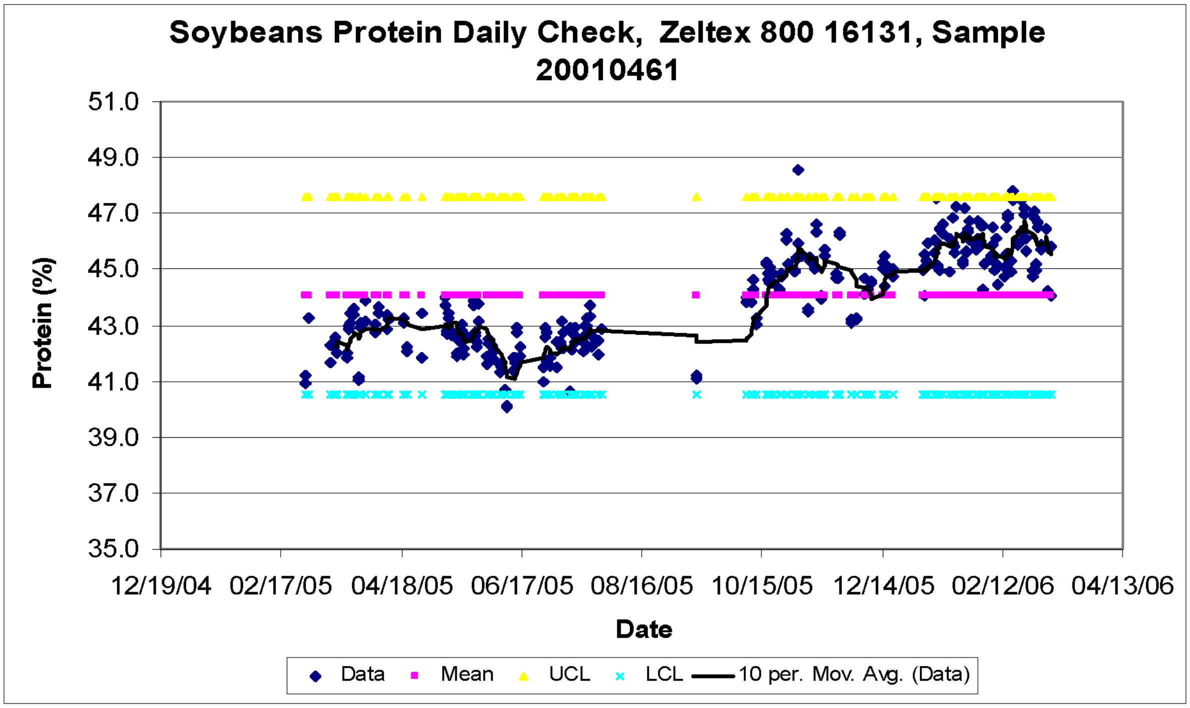


Figure 63. Soybeans Protein Daily Check, Sample 20010461, Zeltex 800 16131 Control Chart

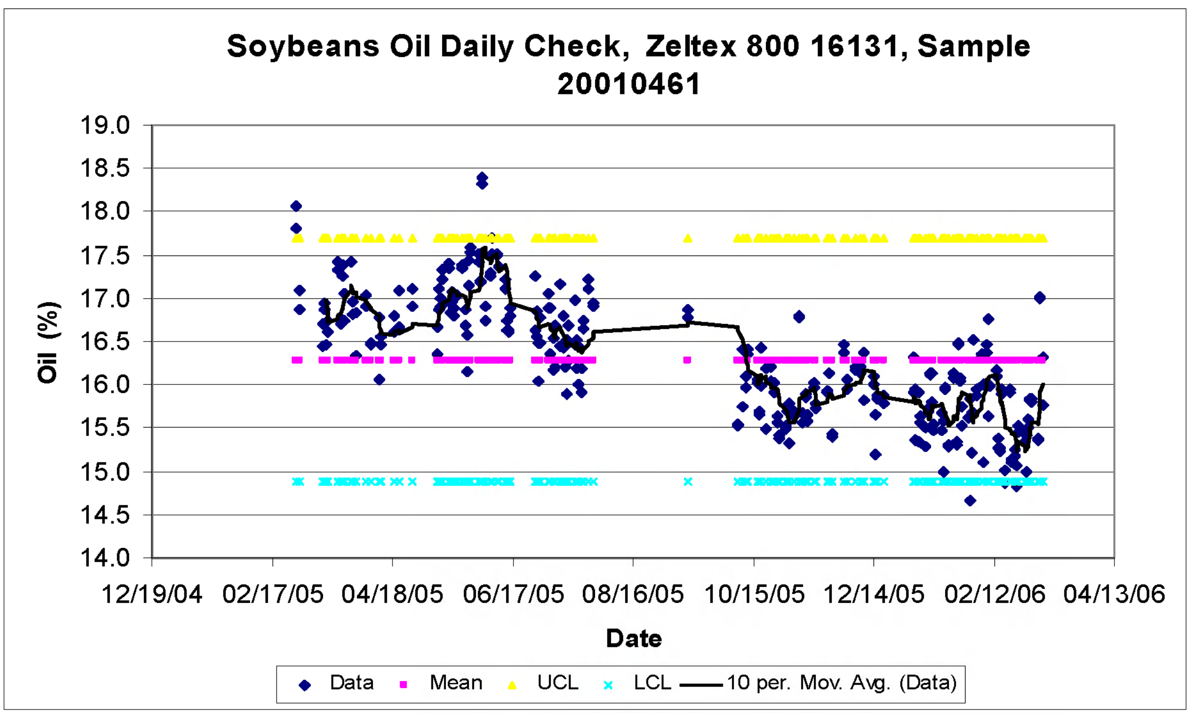


Figure 64. Soybeans Oil Daily Check, Sample 20010461, Zeltex 800 16131 Control Chart

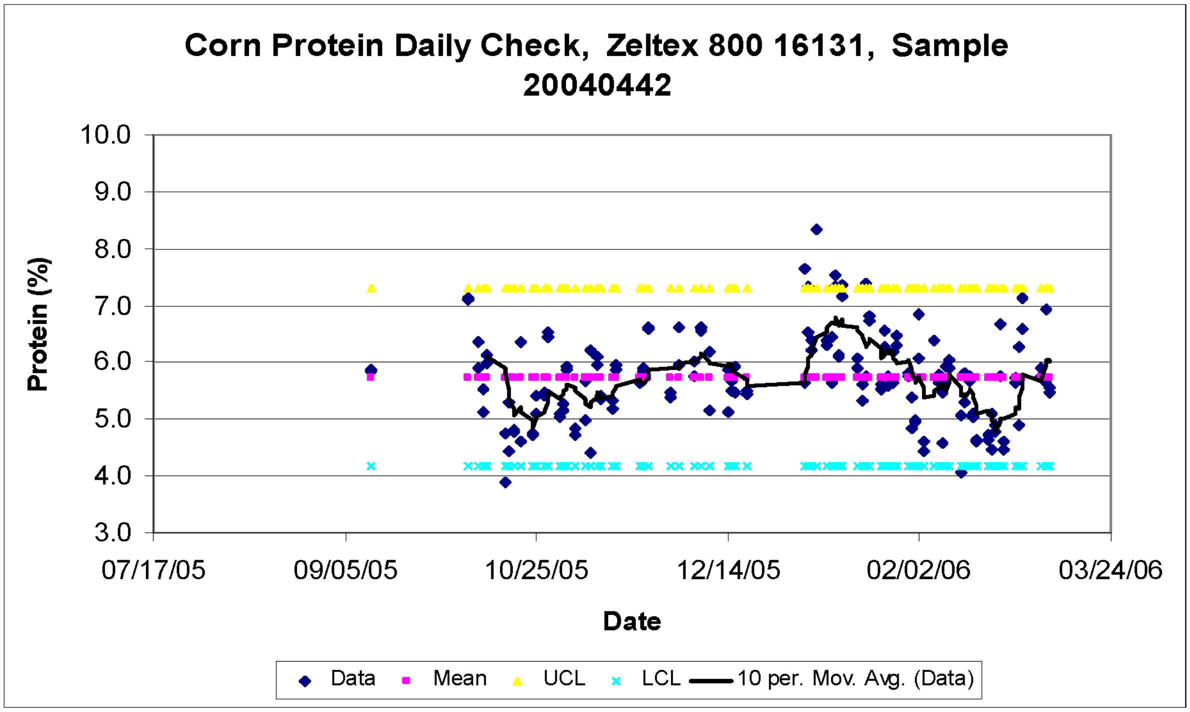


Figure 65. Corn Protein Daily Check, Sample 20040442, Zeltex 800 16131 Control Chart

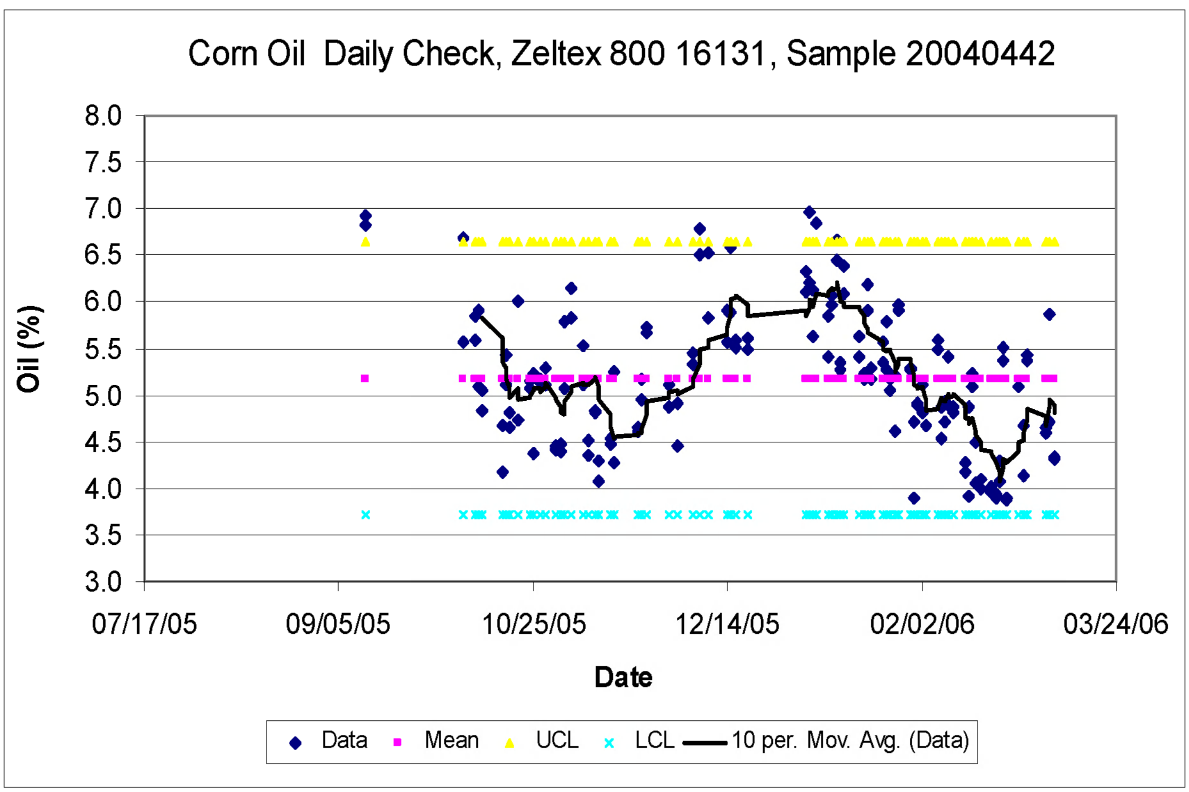


Figure 66. Corn Oil Daily Check, Sample 20040442, Zeltex 800 16131 Control Chart

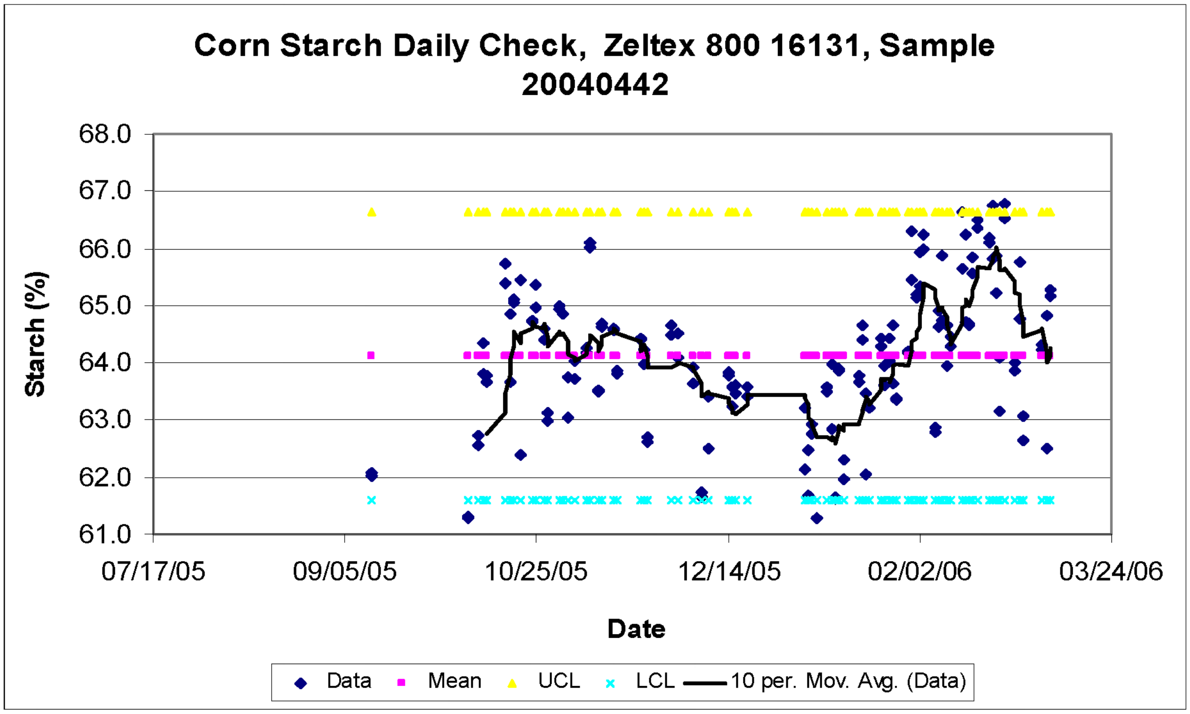


Figure 67. Corn Starch Daily Check, Sample 20040442, Zeltex 800 16131 Control Chart

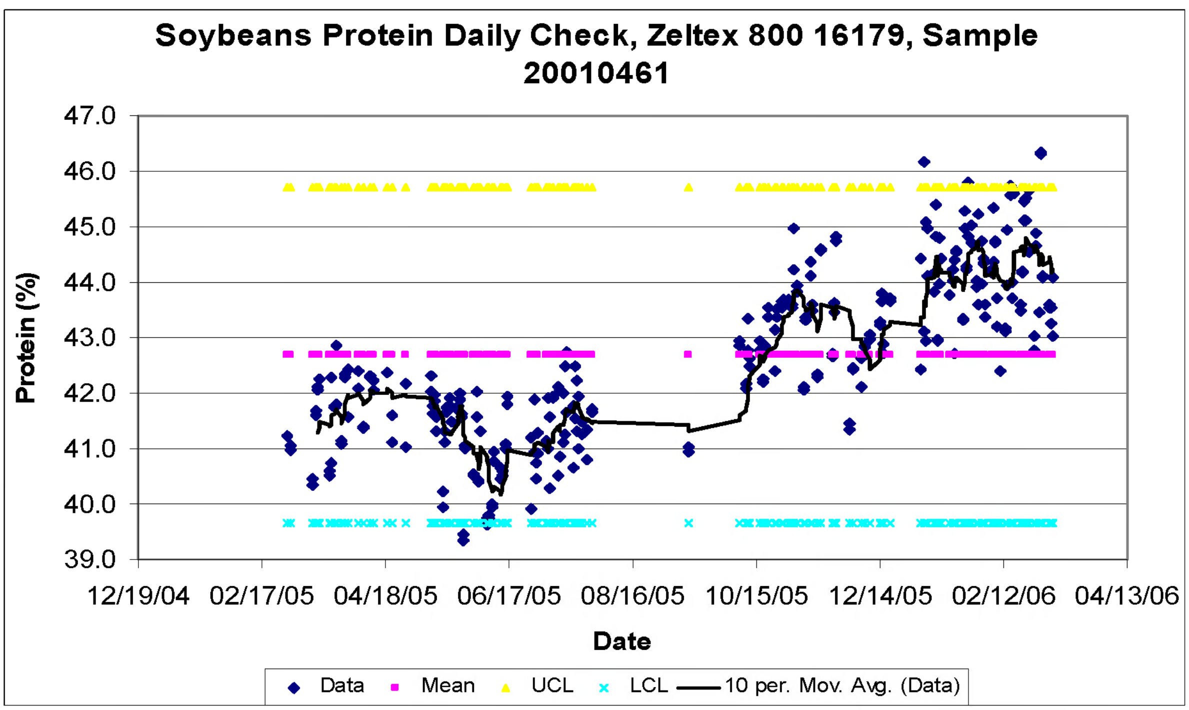


Figure 68. Soybeans Protein Daily Check, Sample 20010461, Zeltex 800 16179 Control Chart

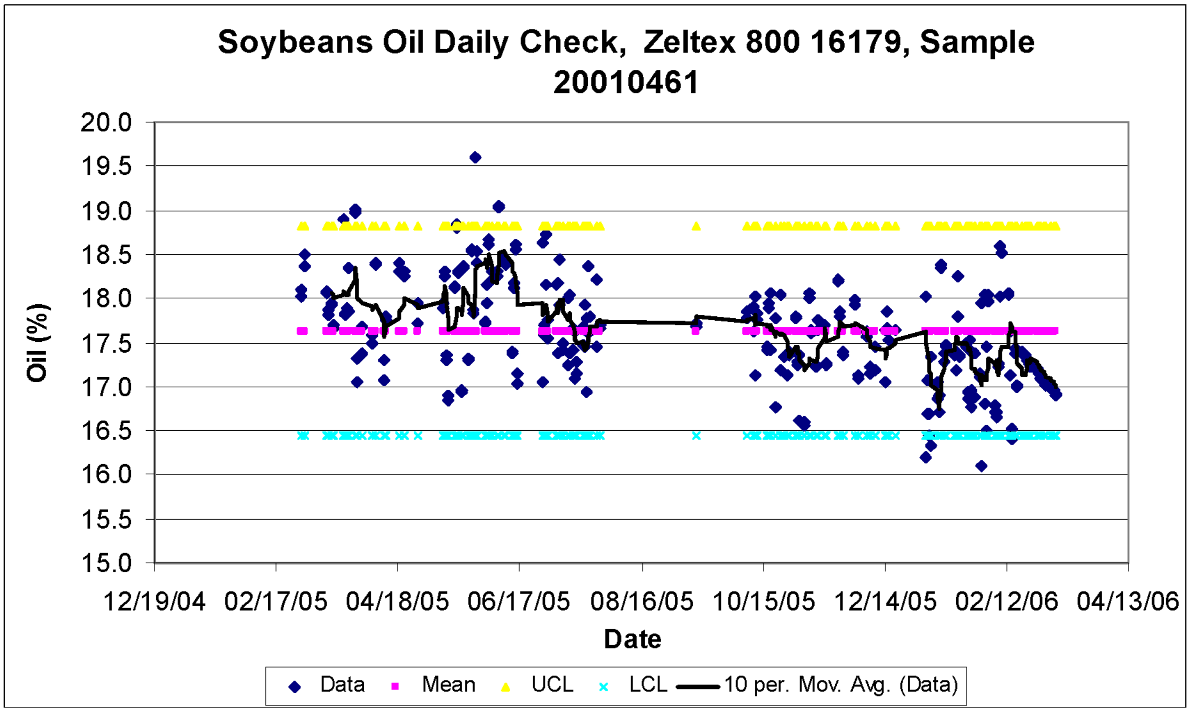


Figure 69. Soybeans Oil Daily Check, Sample 20010461, Zeltex 800 16179 Control Chart

APPENDIX C. NIRS INSTRUMENT DUPLICATES

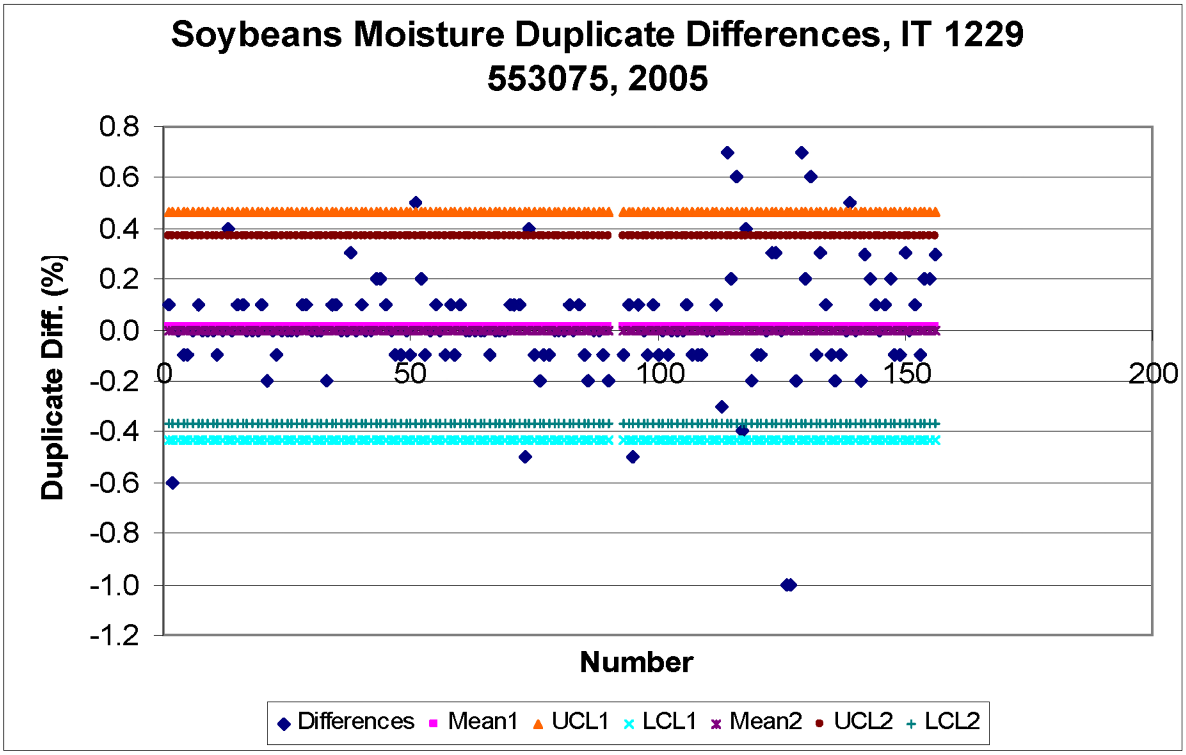


Figure 1. Soybeans Moisture Duplicate Differences 2005, IT 1229 553075 Control Chart, Method 1 and 2

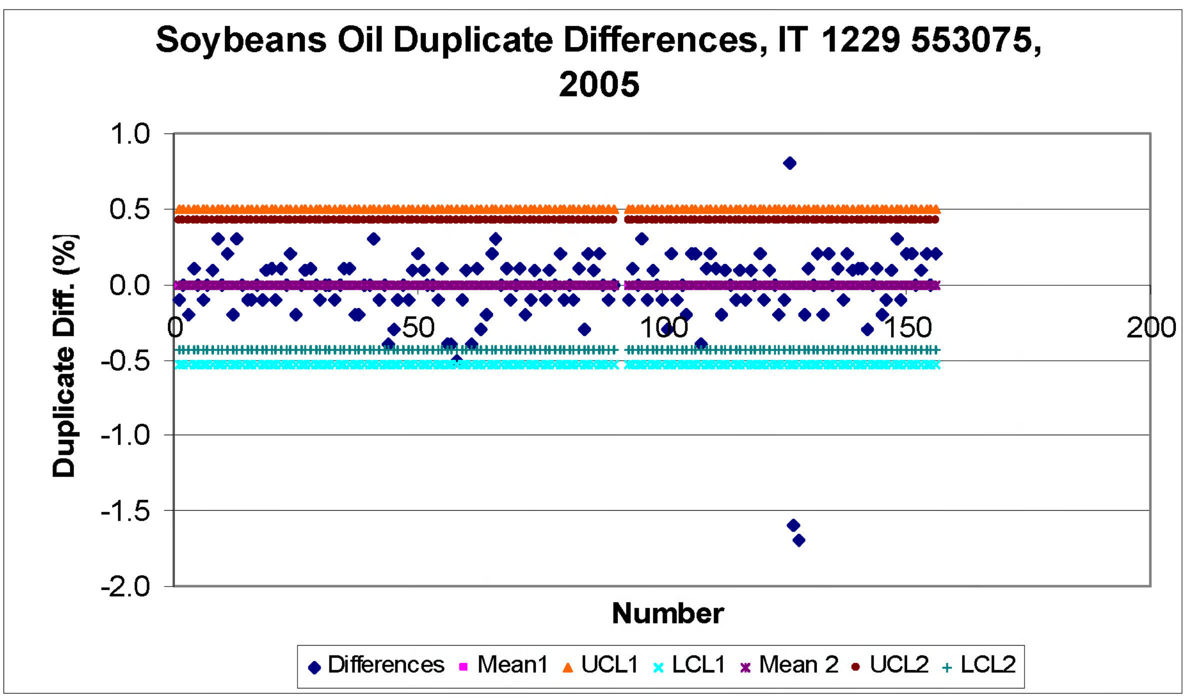


Figure 2. Soybeans Oil Duplicate Differences 2005, IT 1229 553075 Control Chart, Method 1 and 2

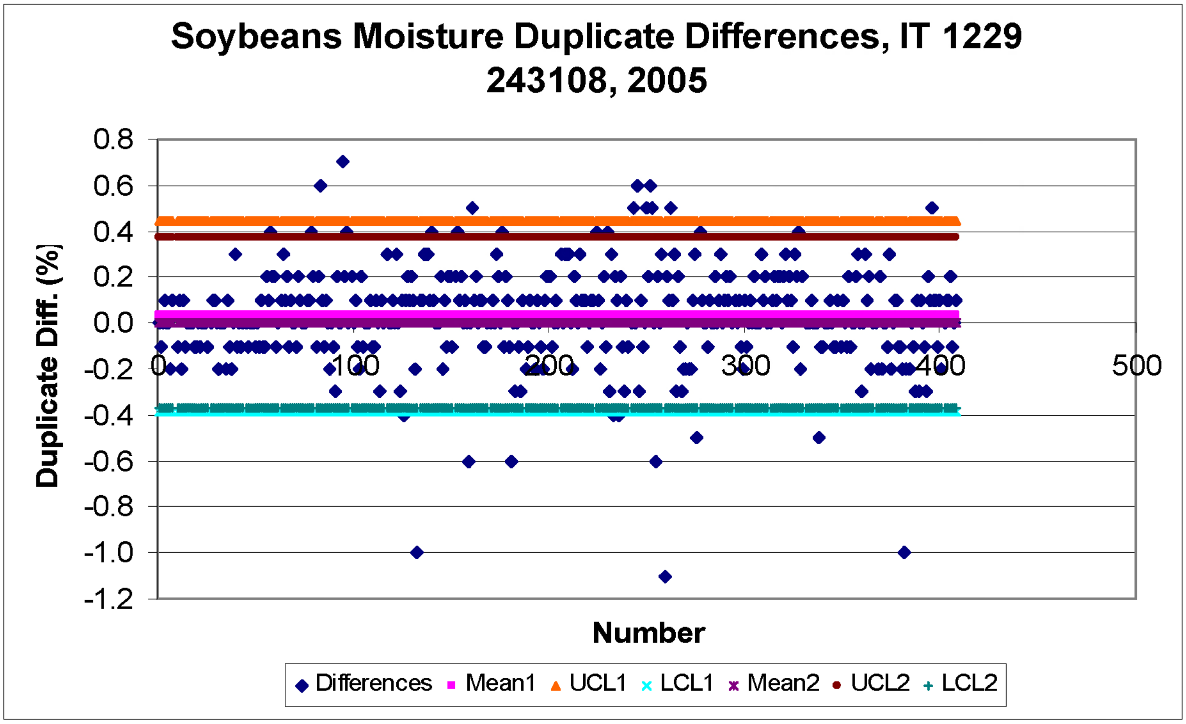


Figure 3. Soybeans Moisture Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

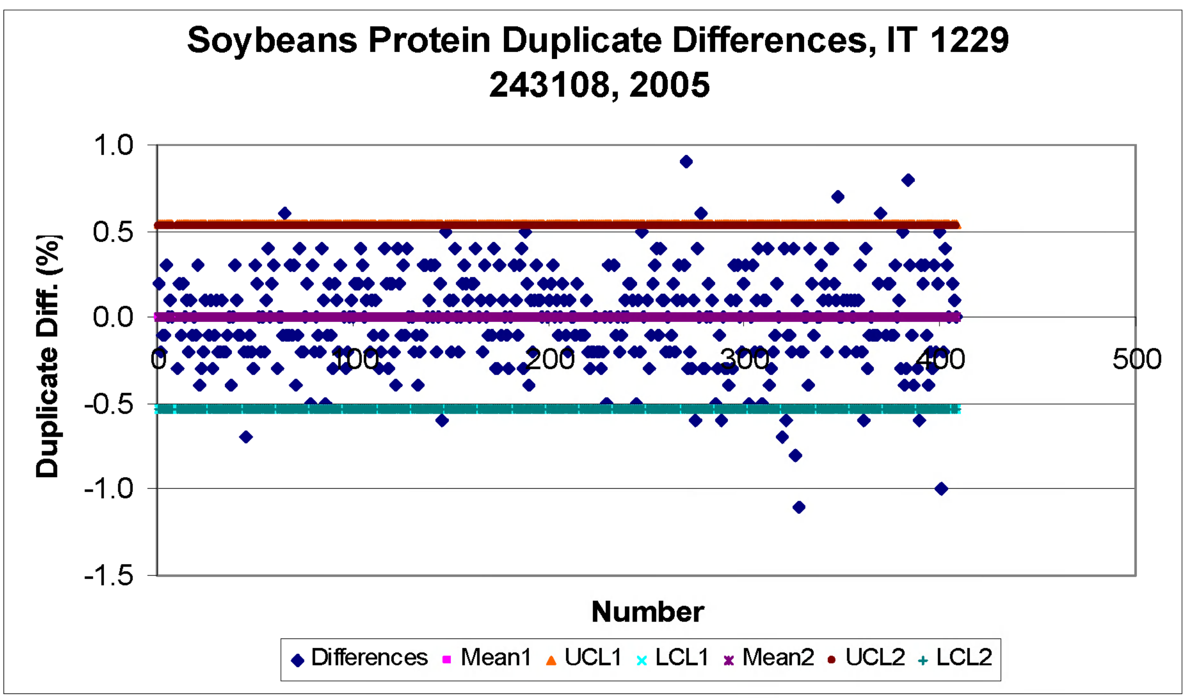


Figure 4. Soybeans Protein Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

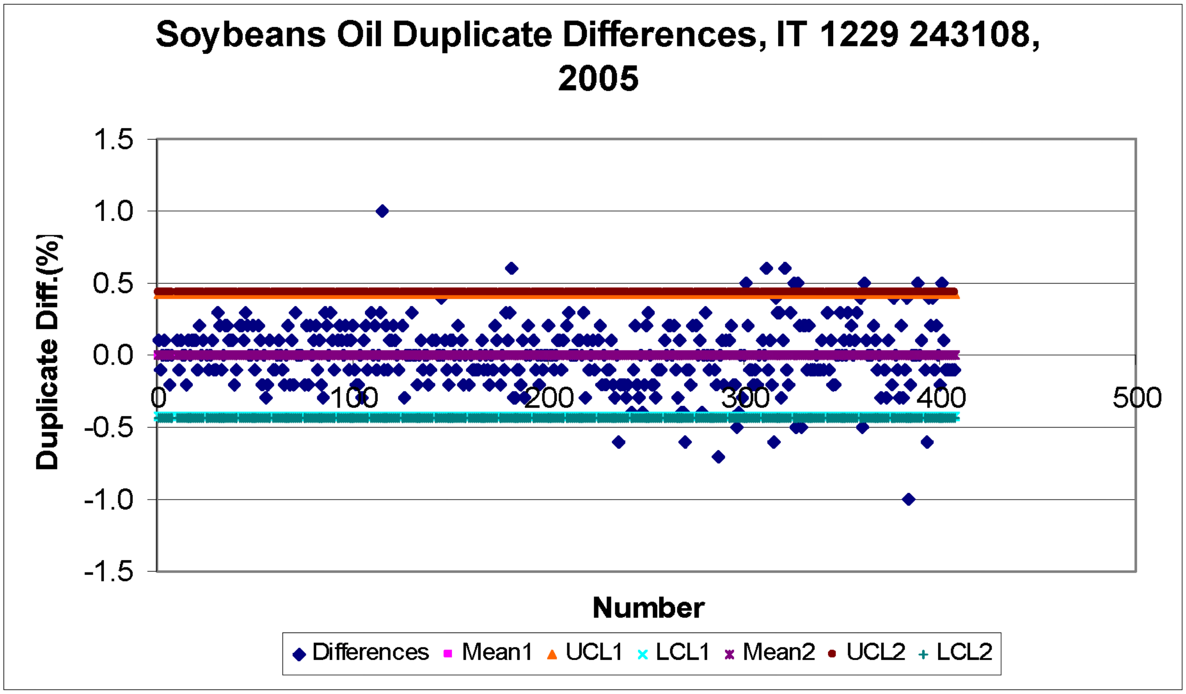


Figure 5. Soybeans Oil Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

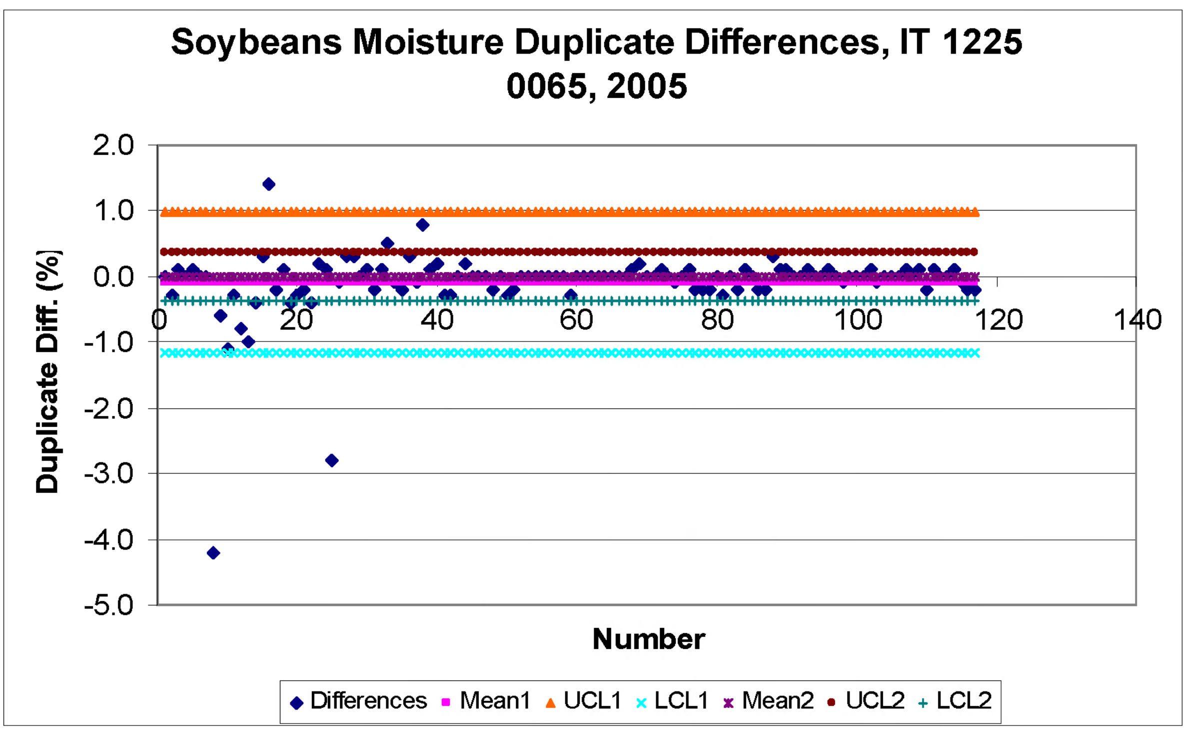


Figure 6. Soybeans Moisture Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

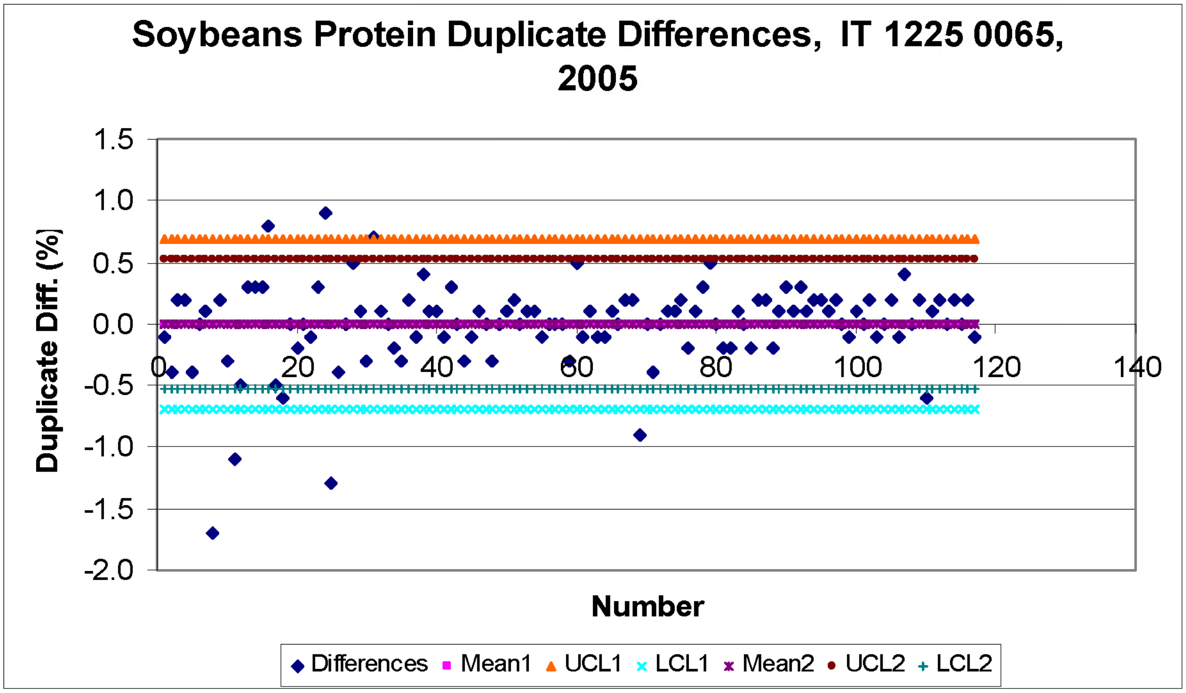


Figure 7. Soybeans Protein Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

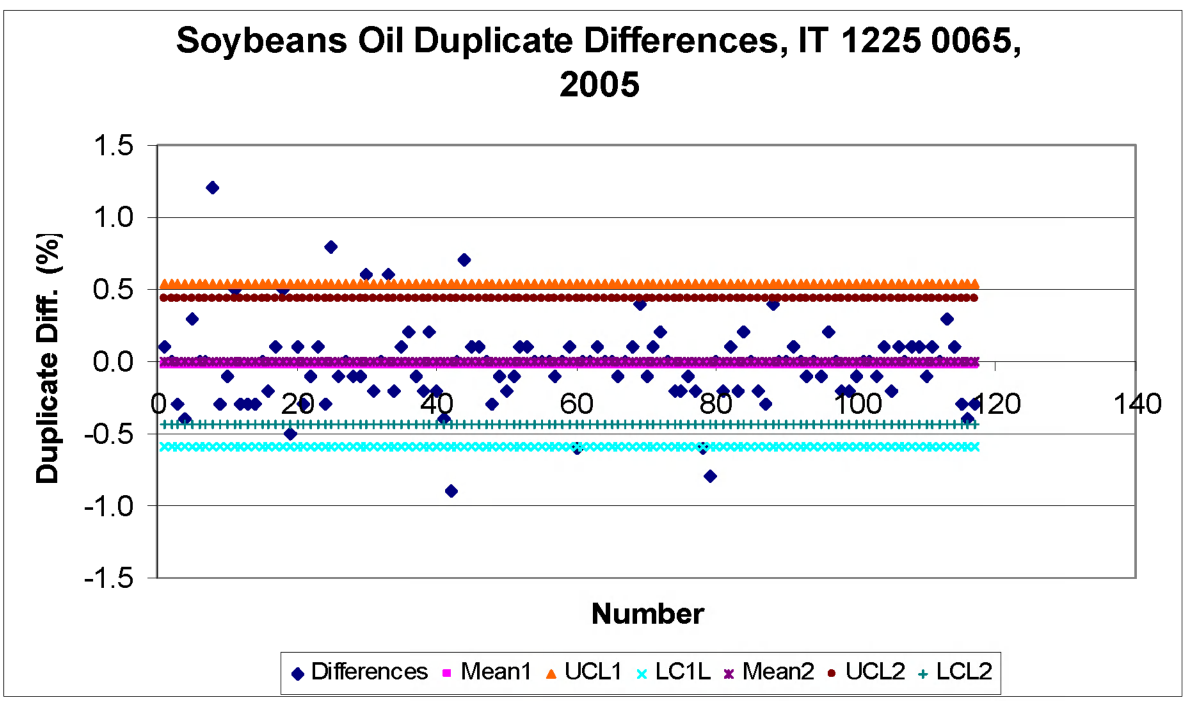


Figure 8. Soybeans Oil Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

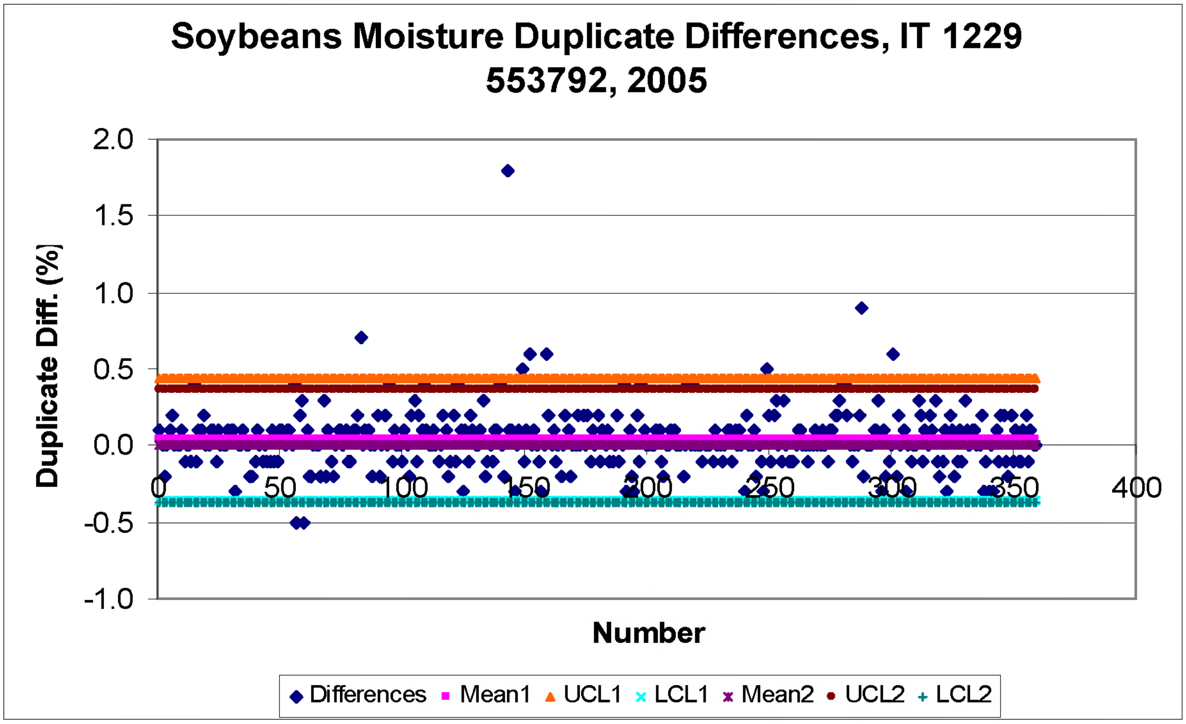


Figure 9. Soybeans Moisture Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

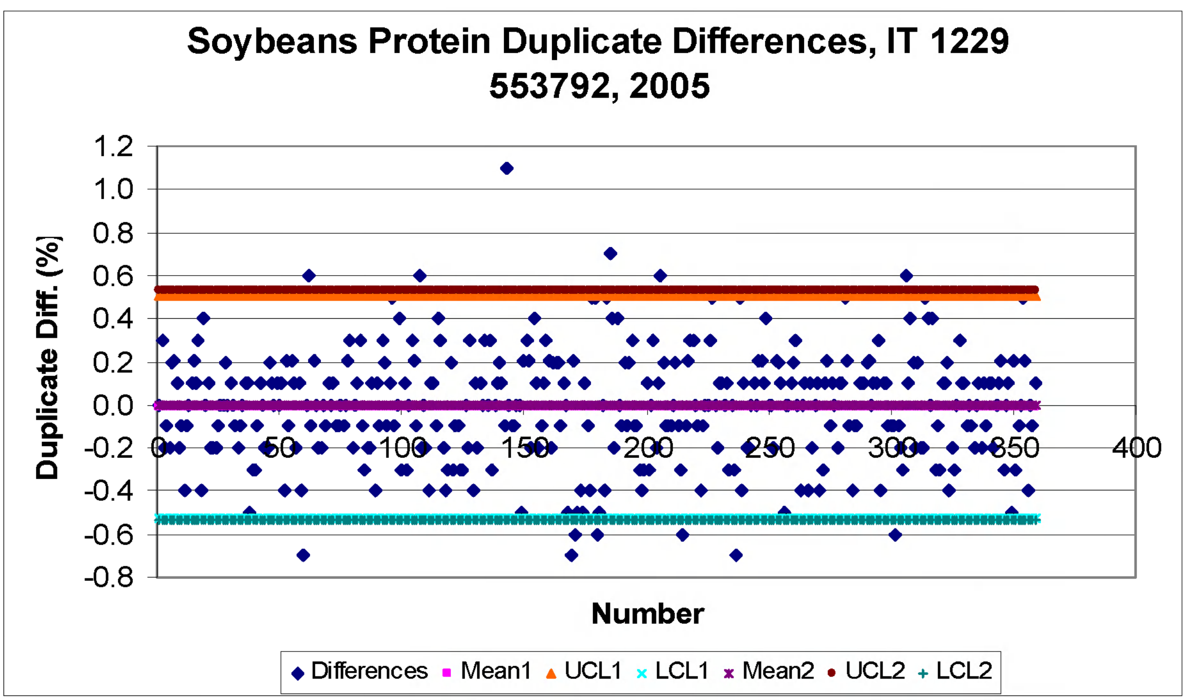


Figure 10. Soybeans Protein Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

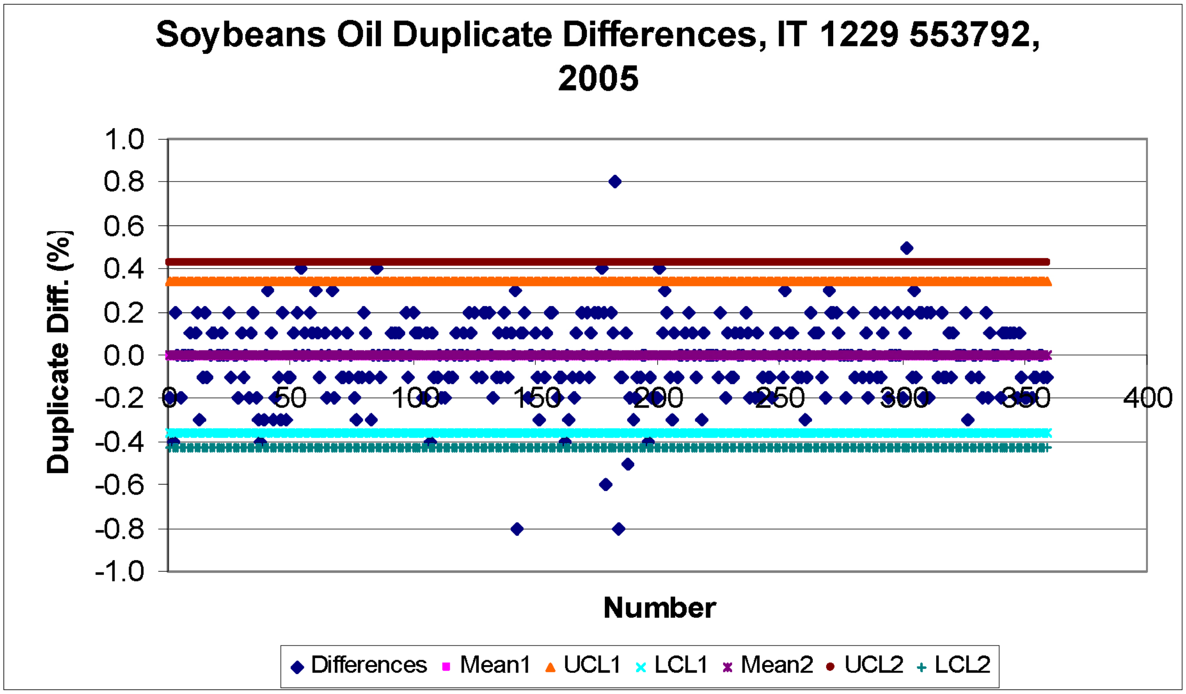


Figure 11. Soybeans Oil Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

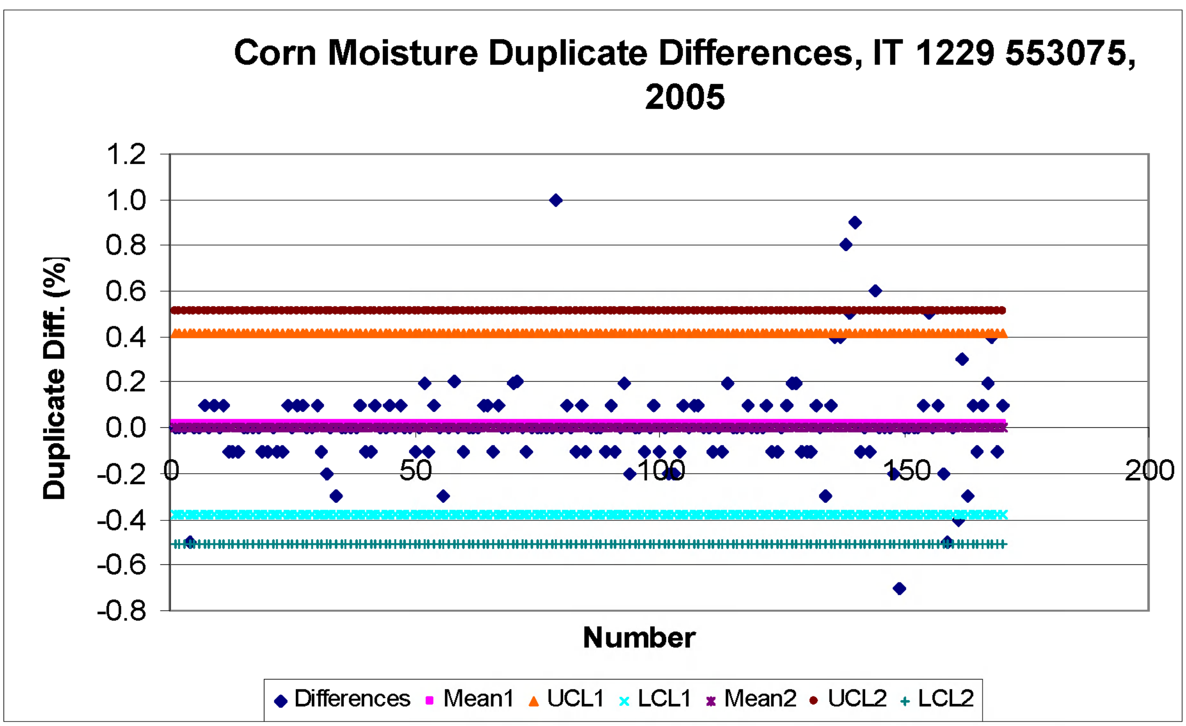


Figure 12. Corn Moisture Duplicate Differences 2005, IT 1229 553075 Control Chart, Method 1 and 2

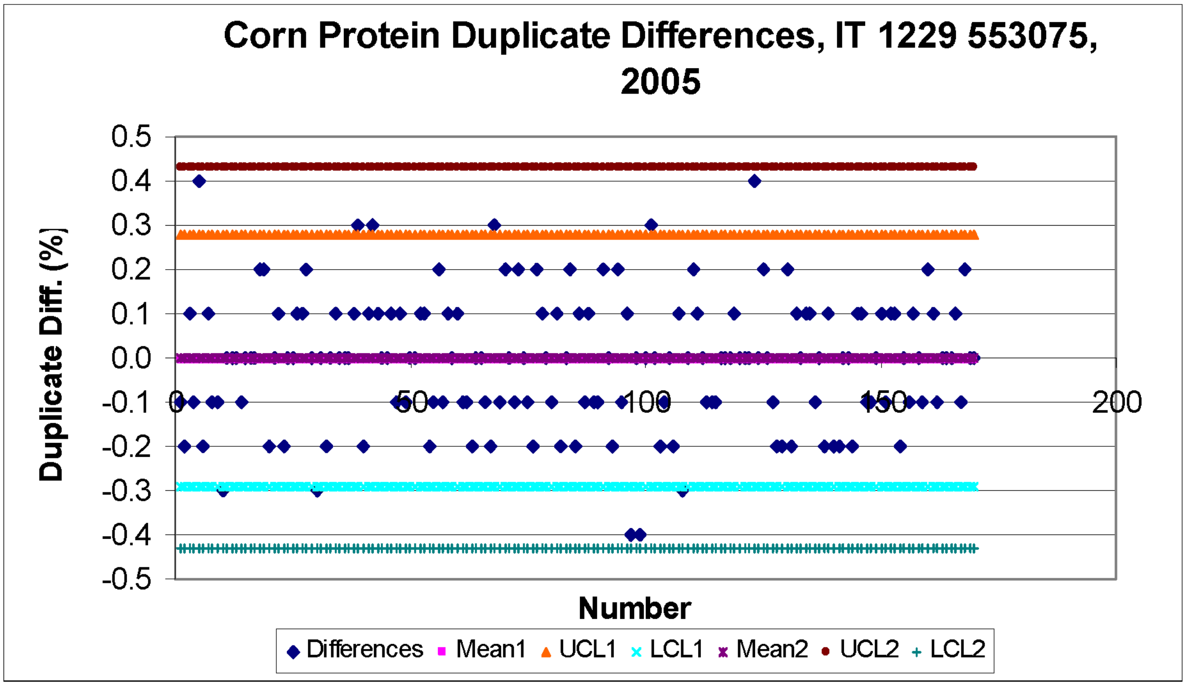


Figure 13. Corn Protein Duplicate Differences 2005, IT 1229 553075 Control Chart, Method 1 and 2

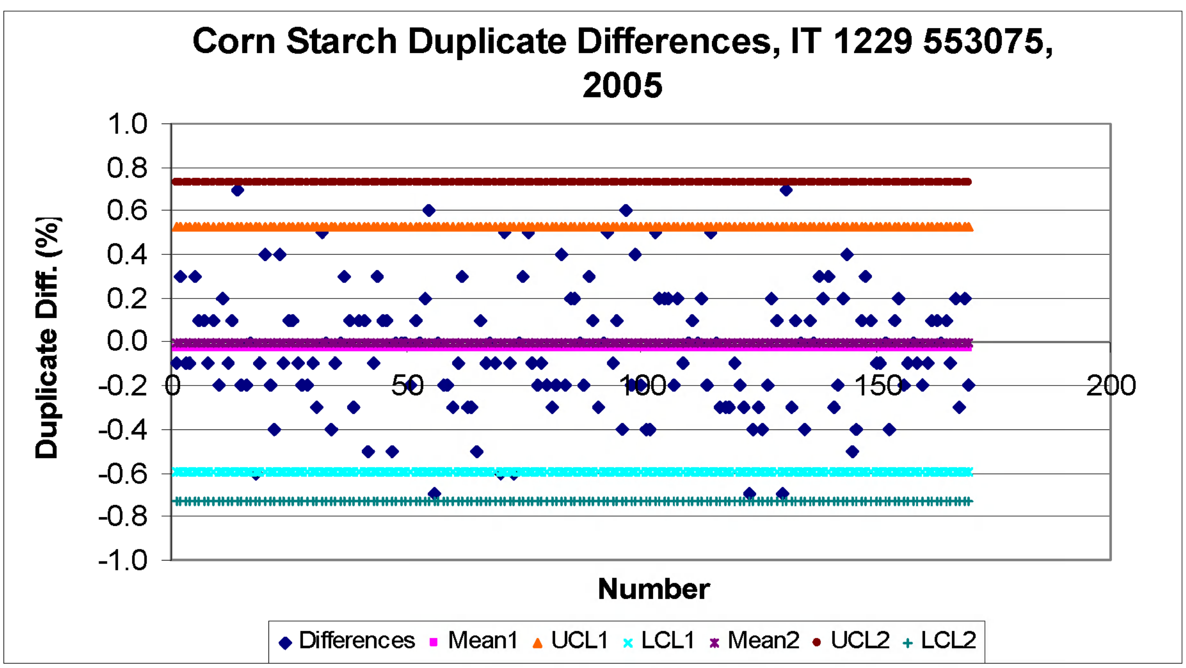


Figure 14. Corn Starch Duplicate Differences 2005, IT 1229 553075 Control Chart, Method 1 and 2

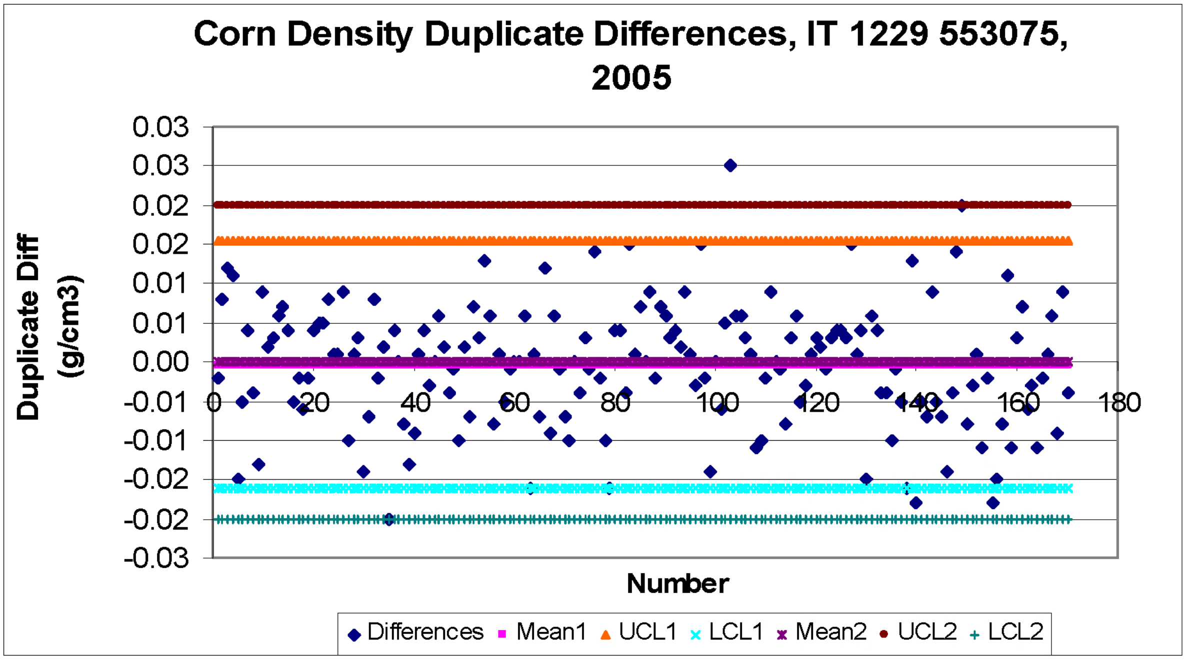


Figure 15. Corn Density Duplicate Differences 2005, IT 1229 553075 Control Chart, Method 1 and 2

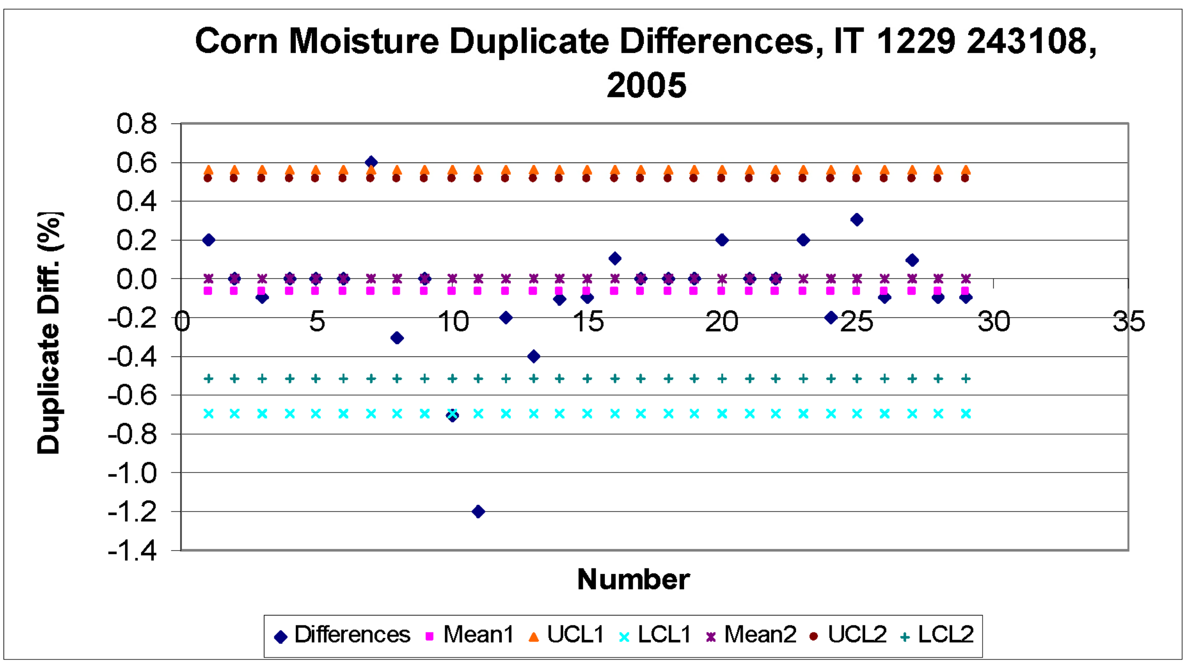


Figure 16. Corn Moisture Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

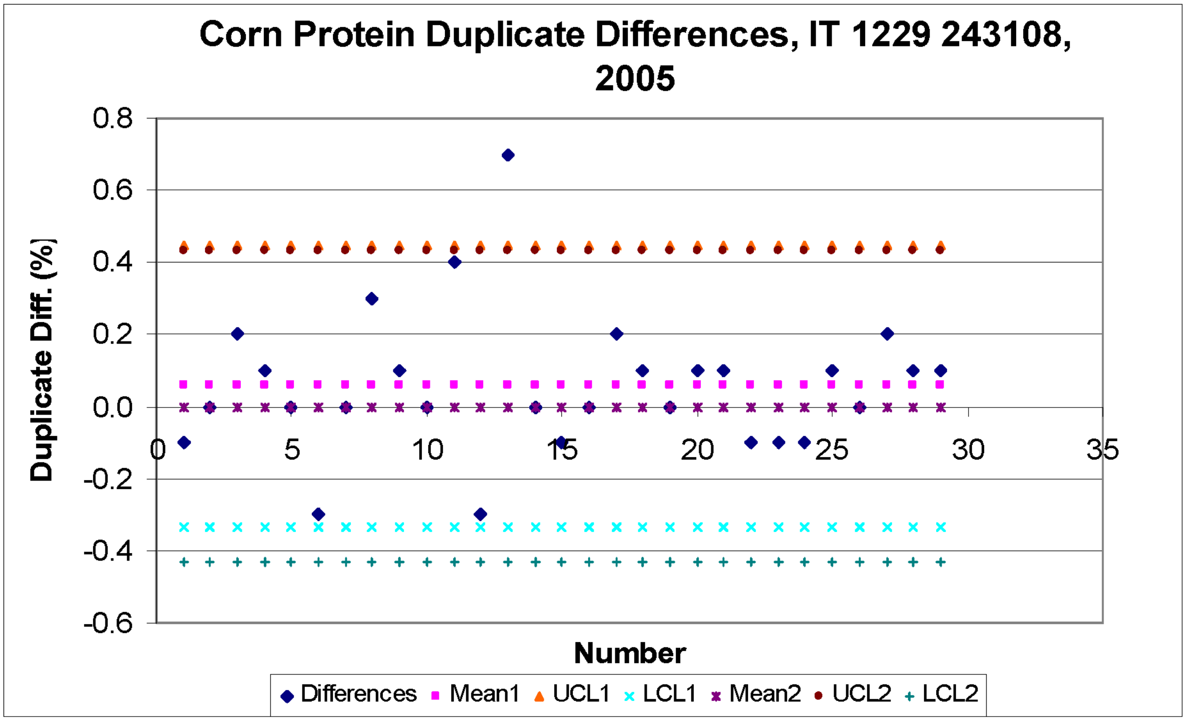


Figure 17. Corn Protein Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

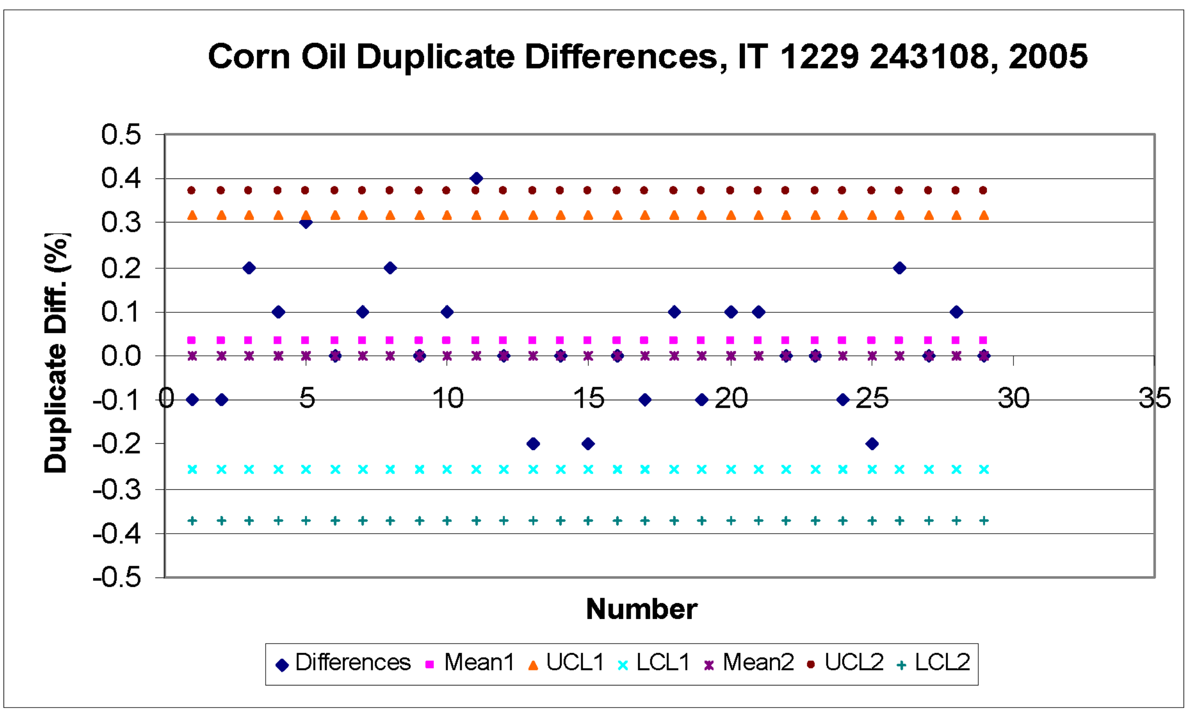


Figure 18. Corn Oil Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

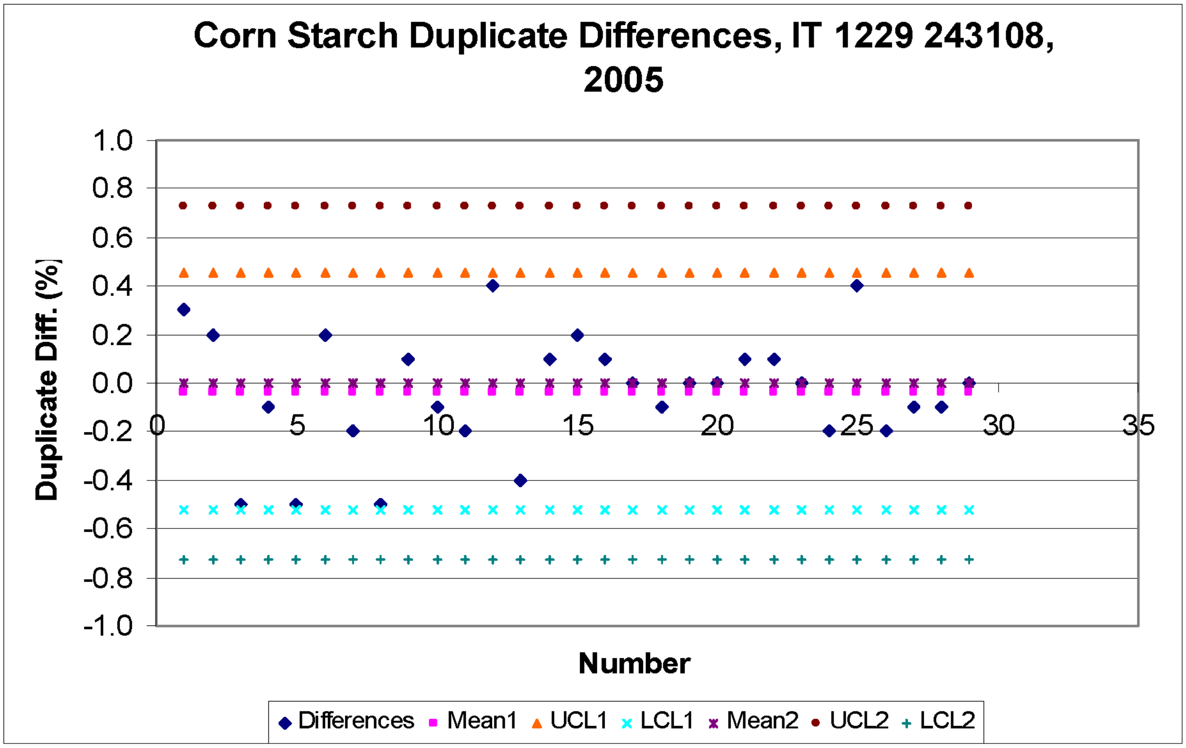


Figure 19. Corn Starch Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

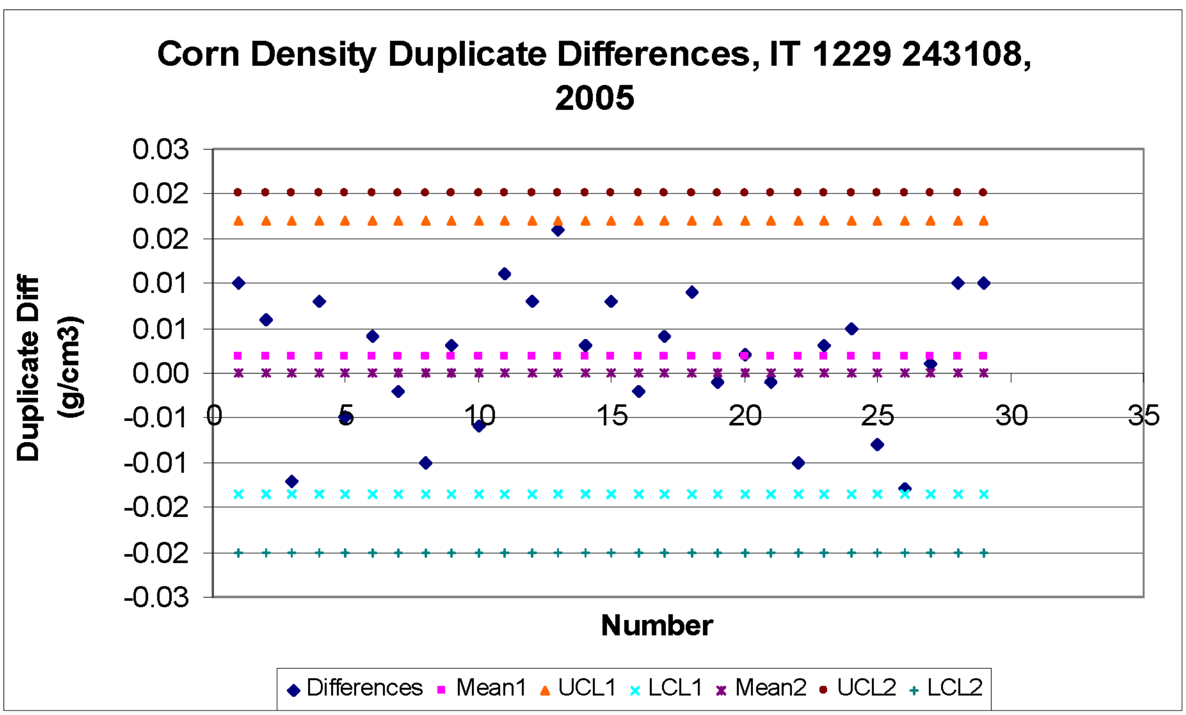


Figure 20. Corn Density Duplicate Differences 2005, IT 1229 243108 Control Chart, Method 1 and 2

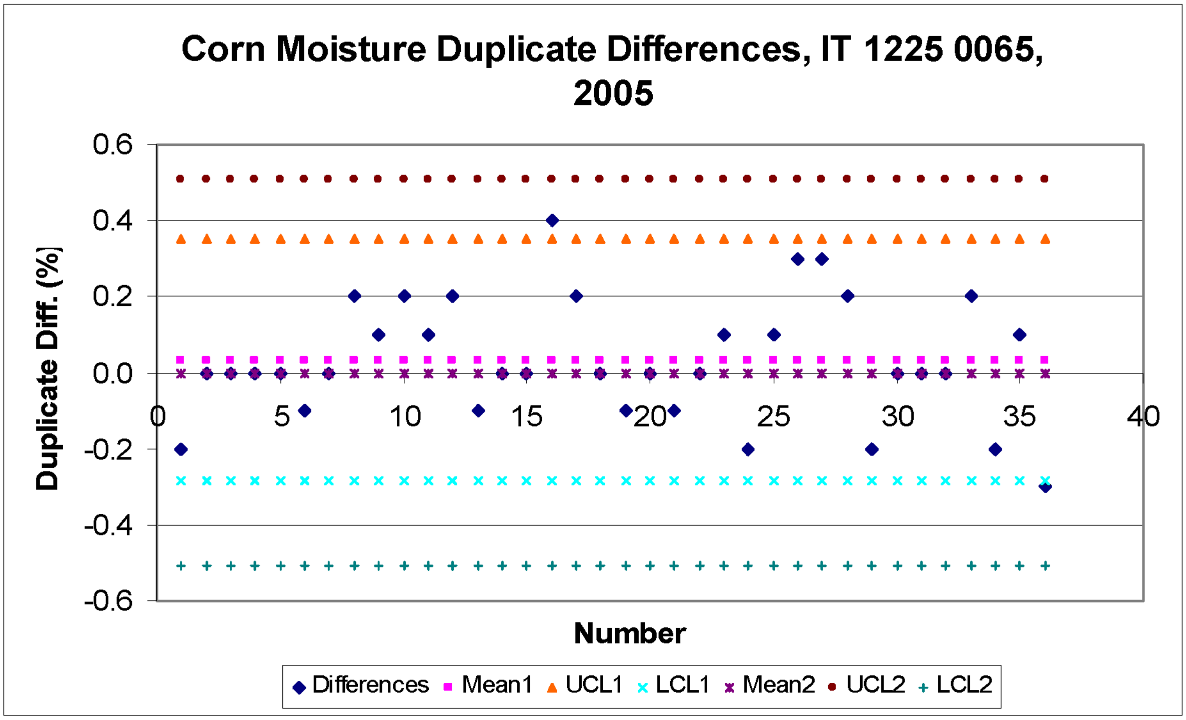


Figure 21. Corn Moisture Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

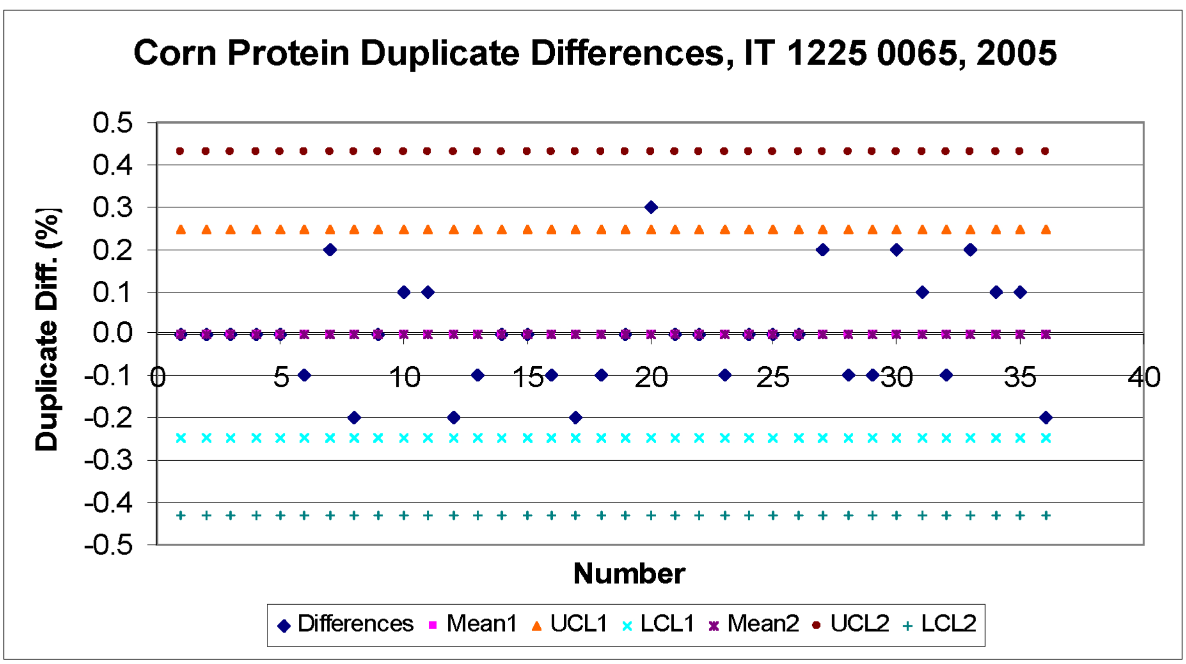


Figure 22. Corn Protein Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

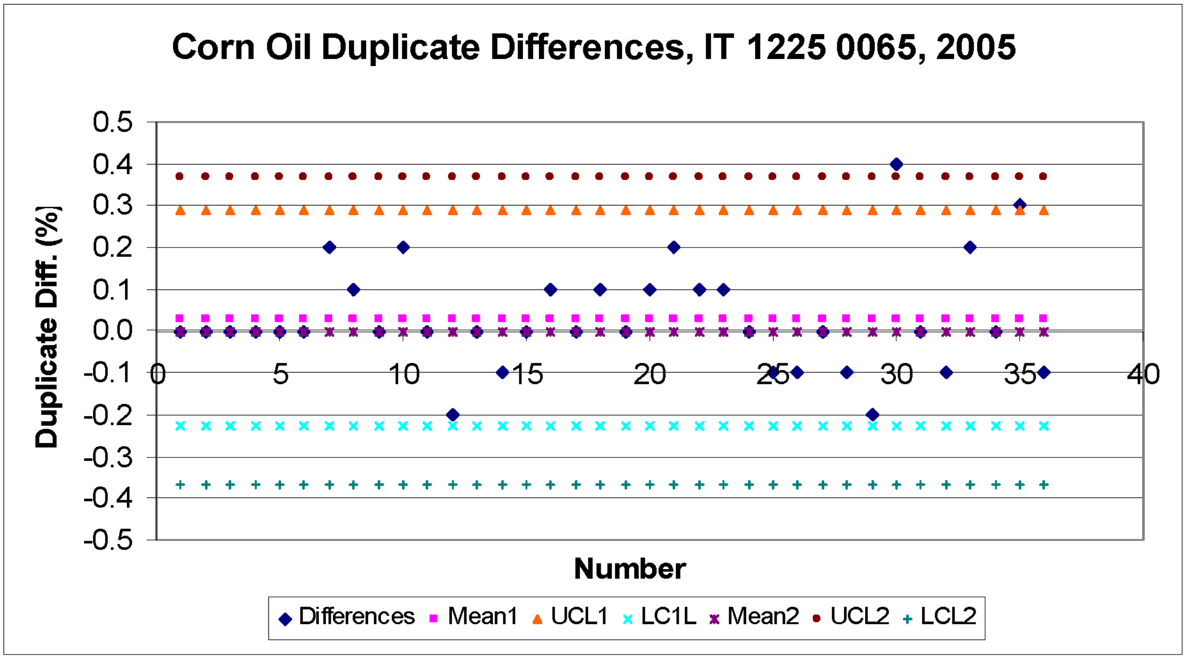


Figure 23. Corn Oil Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

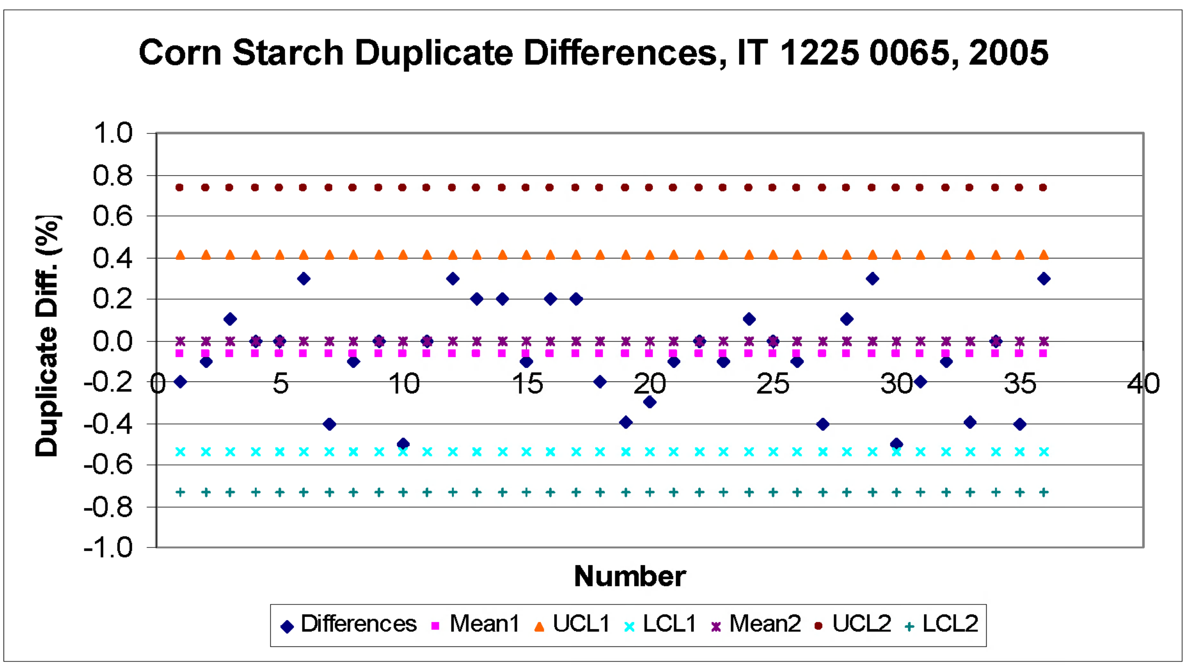


Figure 24. Corn Starch Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

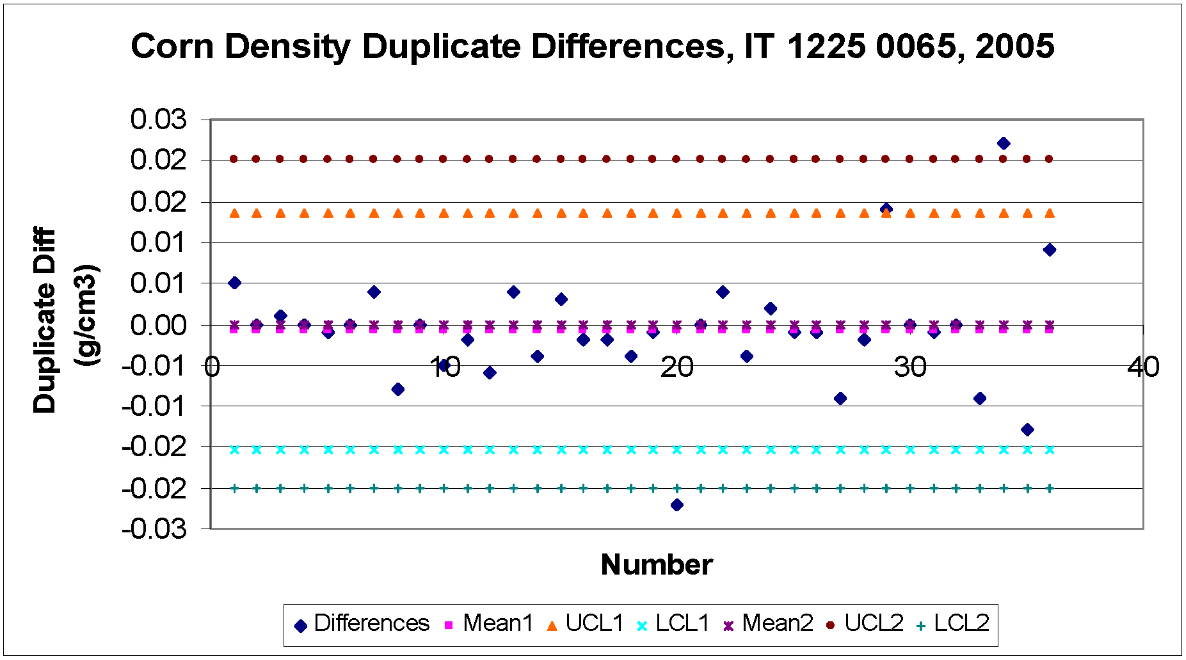


Figure 25. Corn Density Duplicate Differences 2005, IT 1225 0065 Control Chart, Method 1 and 2

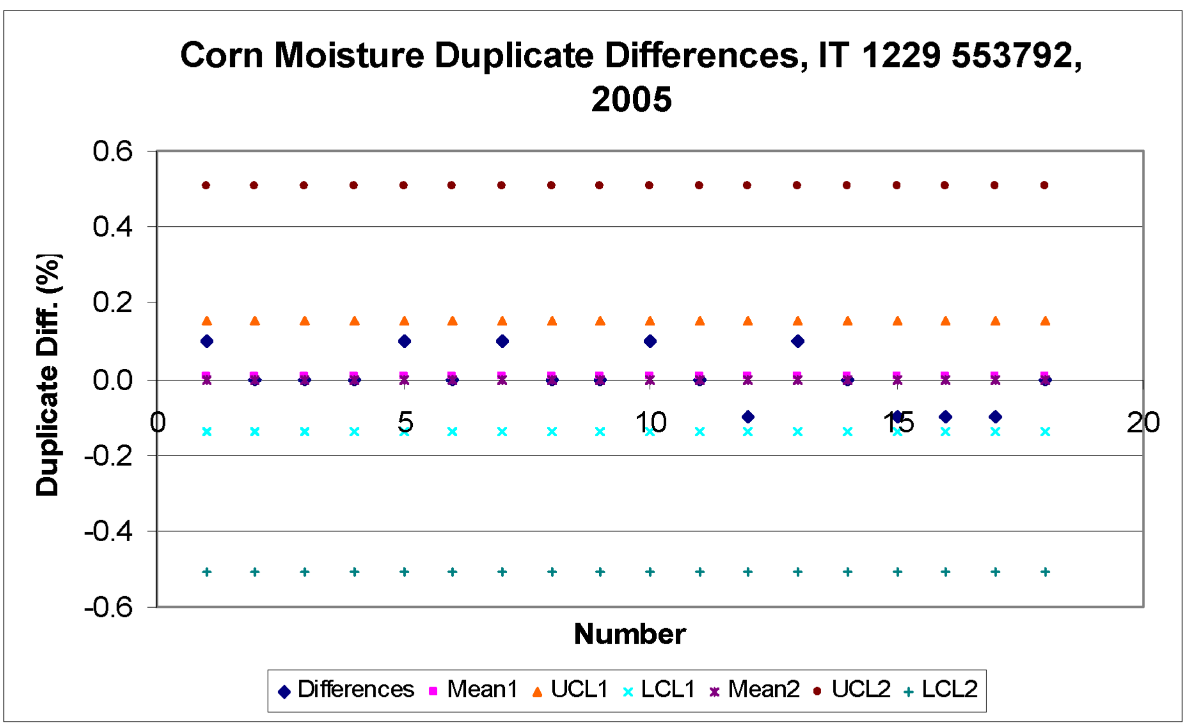


Figure 26. Corn Moisture Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

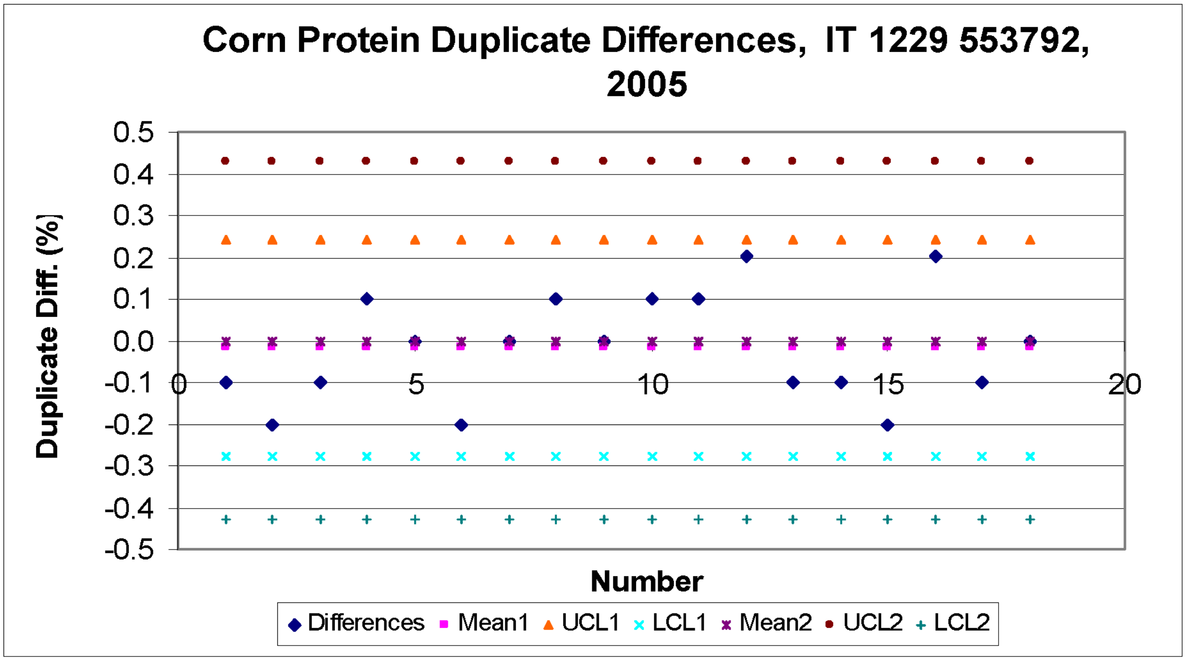


Figure 27. Corn Protein Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

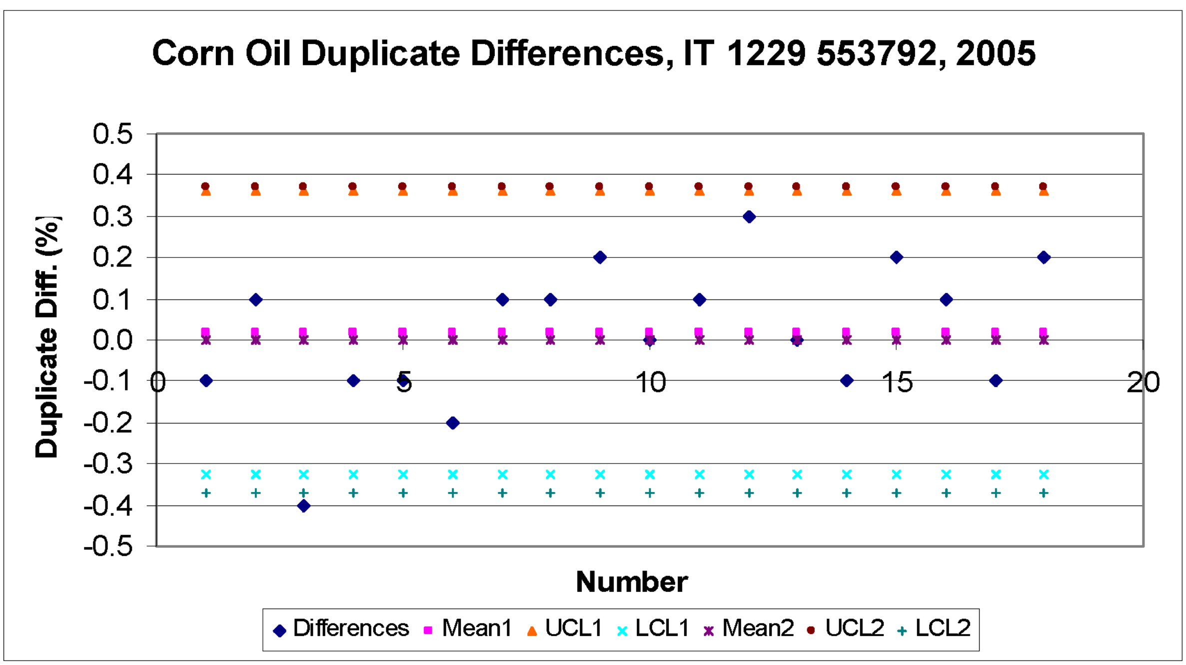


Figure 28. Corn Oil Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

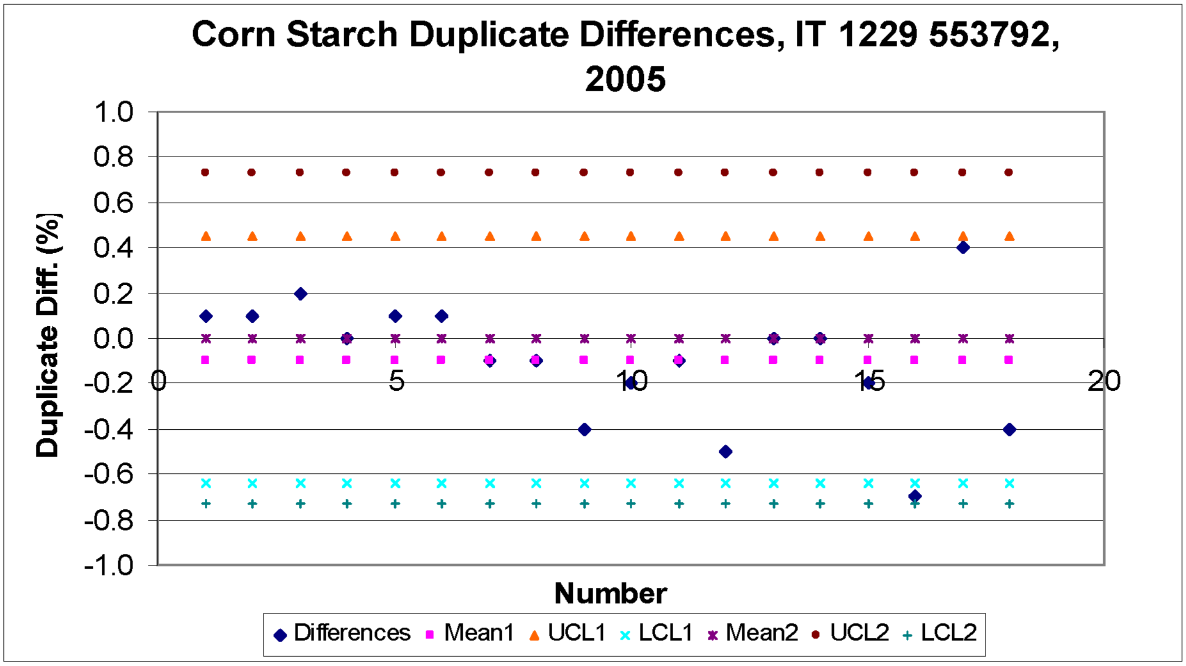


Figure 29. Corn Starch Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

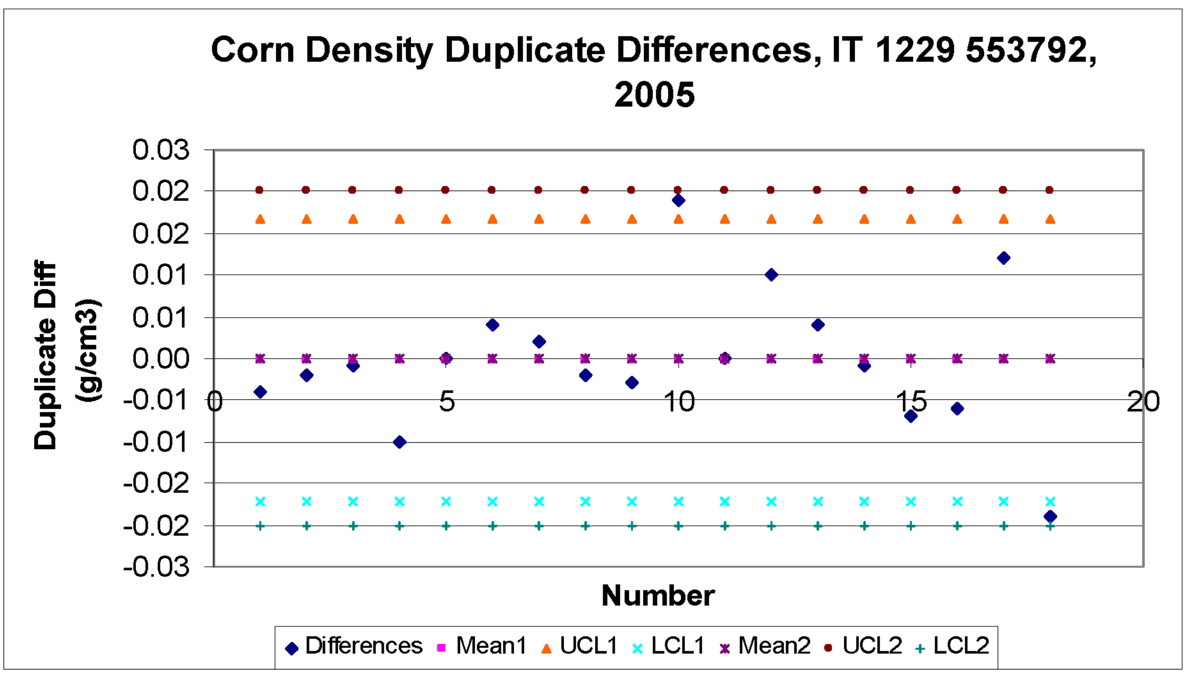


Figure 30. Corn Density Duplicate Differences 2005, IT 1229 553792 Control Chart, Method 1 and 2

Table 1. NIRS Instrument Duplicate Differences Summary, Soybeans 2004

Factor	NIR Unit	Average of Differences	Standard Deviation of Differences	n	Out of control	
					Method 1	Method 2
Moisture (%)	IT553075	0.073	0.447	216	5.56%	24.07%
	IT243108	0.017	0.134	29	6.90%	3.45%
	IT65	-0.072	0.330	25	8.00%	20.00%
	IT 553792	0.009	0.177	257	4.67%	4.67%
Protein (%)	IT553075	-0.030	0.388	216	5.56%	14.35%
	IT243108	-0.093	0.212	29	6.90%	3.45%
	IT65	-0.076	0.194	25	12.00%	4.00%
	IT 553792	-0.046	0.370	257	7.00%	12.06%
Oil (%)	IT553075	-0.021	0.252	216	7.41%	9.72%
	IT243108	0.034	0.149	29	6.90%	0.00%
	IT65	-0.024	0.213	25	8.00%	8.00%
	IT 553792	0.019	0.185	257	5.45%	2.33%
Average					**7.03%**	**8.84%**

Table 2. NIRS Instrument Duplicate Differences Summary, Corn 2004

Factor	NIR Unit	Average of Differences	Standard Deviation of Differences	n	Out of control	
					Method 1	Method 2
Moisture (%)	IT553075	0.00	0.66	201	2.49%	8.96%
	IT243108	-0.01	0.35	355	2.82%	3.94%
Protein (%)	IT553075	0.02	0.22	201	6.47%	6.47%
	IT243108	-0.02	0.28	355	3.38%	5.07%
Oil (%)	IT553075	0.01	0.23	201	4.98%	9.45%
	IT243108	0.00	0.16	355	3.10%	3.10%
Starch (%)	IT553075	0.02	0.43	201	5.97%	9.45%
	IT243108	0.00	0.32	355	4.51%	3.10%
Density (g/cm^3)	IT553075	0.001	0.011	201	2.99%	4.98%
	IT243108	0.007	0.095	355	5.37%	5.37%
Average					**4.21%**	**5.99%**

APPENDIX D. ORIGINAL NIRS VS. REFERENCES

Table 1. Original NIRS vs. Reference Chemistry Summary, 2004

	Instrument	N	R^2	Standard Error	Out of Control
Soybeans Protein	IT 1229 553792	32	0.87	0.90	18.75%
	IT 1229 553705	64	0.77	1.84	57.81%
	IT 1225 0065	12	0.99	0.35	0.00%
Soybeans Oil	IT 1229 553792	32	0.86	0.53	9.38%
	IT 1229 553705	64	0.77	0.81	20.31%
	IT 1225 0065	12	0.96	0.26	0.00%
Corn Protein	IT 1229 243108	13	0.83	0.45	7.69%
	IT 1229 553075	49	0.45	0.65	24.49%
Corn Oil	IT 1229 243108	13	0.07	0.27	7.69%
	IT 1229 553075	49	0.16	0.30	14.29%
Corn Starch	IT 1229 243108	13	0.33	0.52	15.38%
	IT 1229 553075	49	0.35	0.60	18.37%
Corn Density	IT 1229 243108	13	0.48	0.01	7.69%
	IT 1229 553075	49	0.03	0.02	28.57%

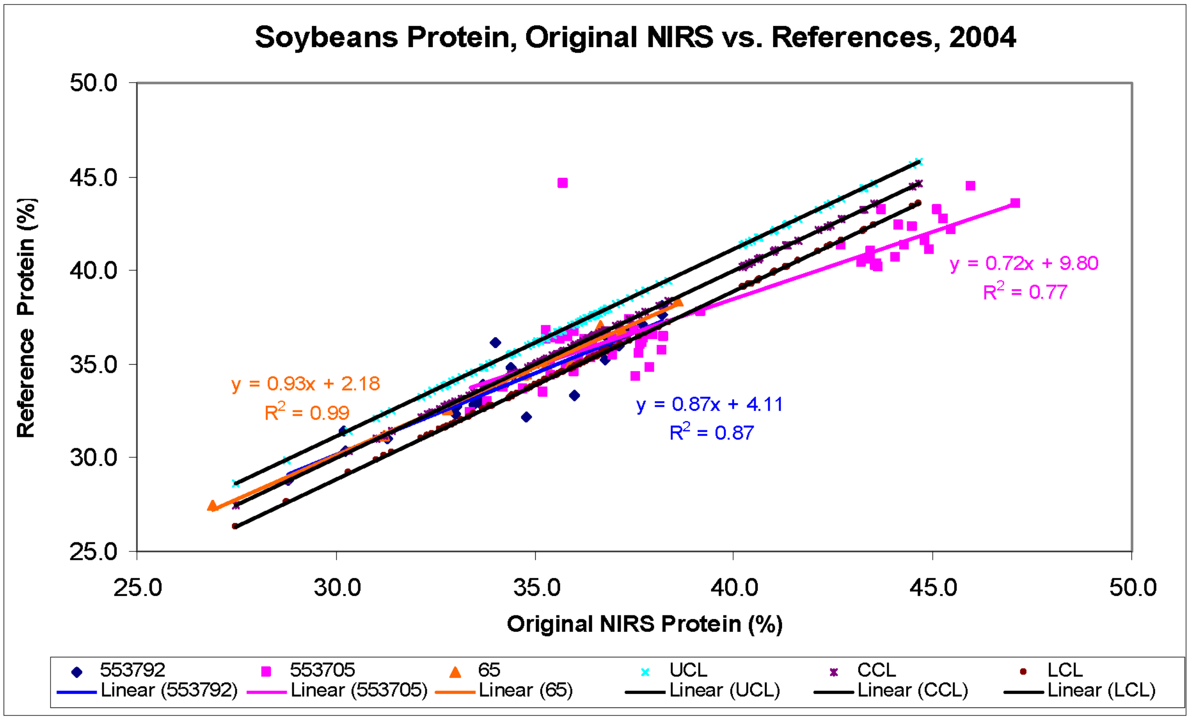


Figure 1. Soybeans Protein, Original NIRS vs. References, 2004

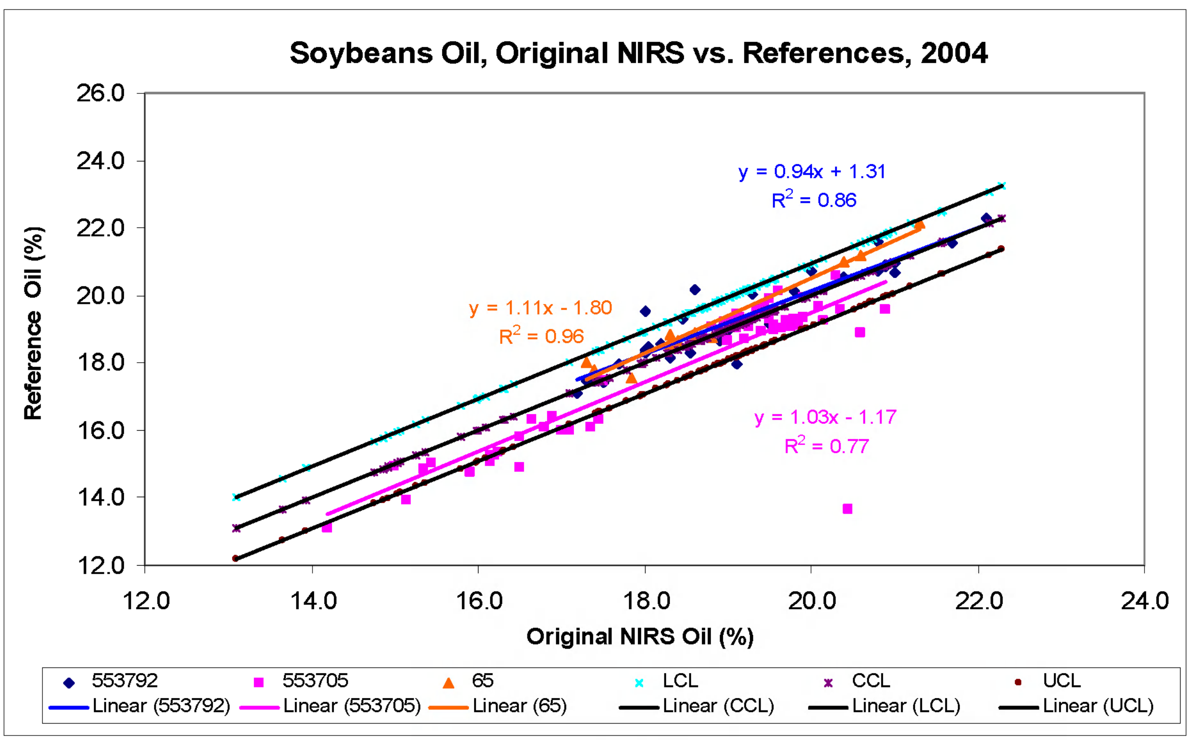


Figure 2. Soybeans Oil, Original NIRS vs. References, 2004

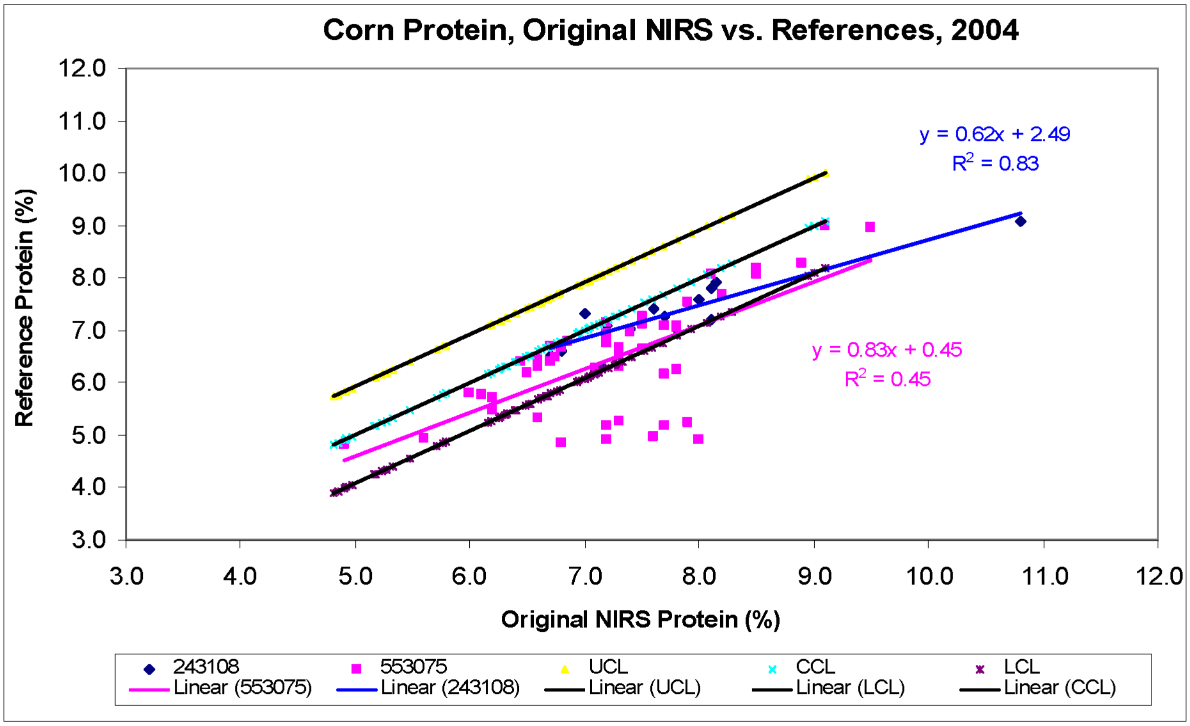


Figure 3. Corn Protein, Original NIRS vs. References, 2004

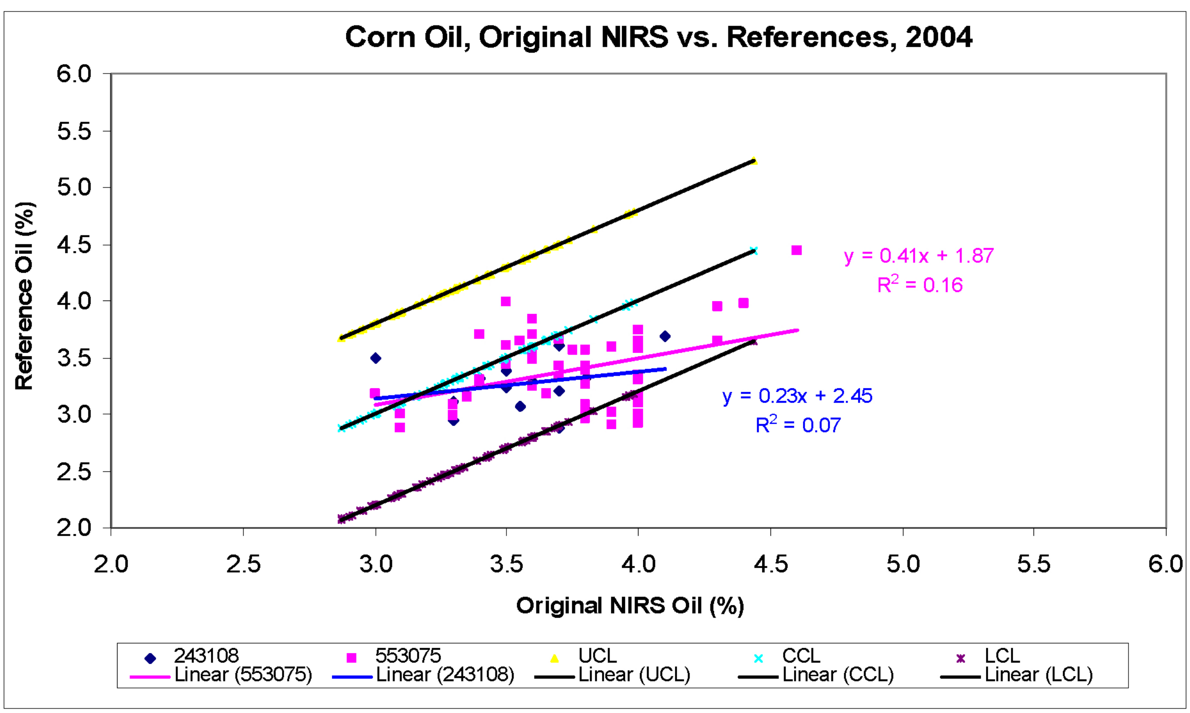


Figure 4. Corn Oil, Original NIRS vs. References, 2004

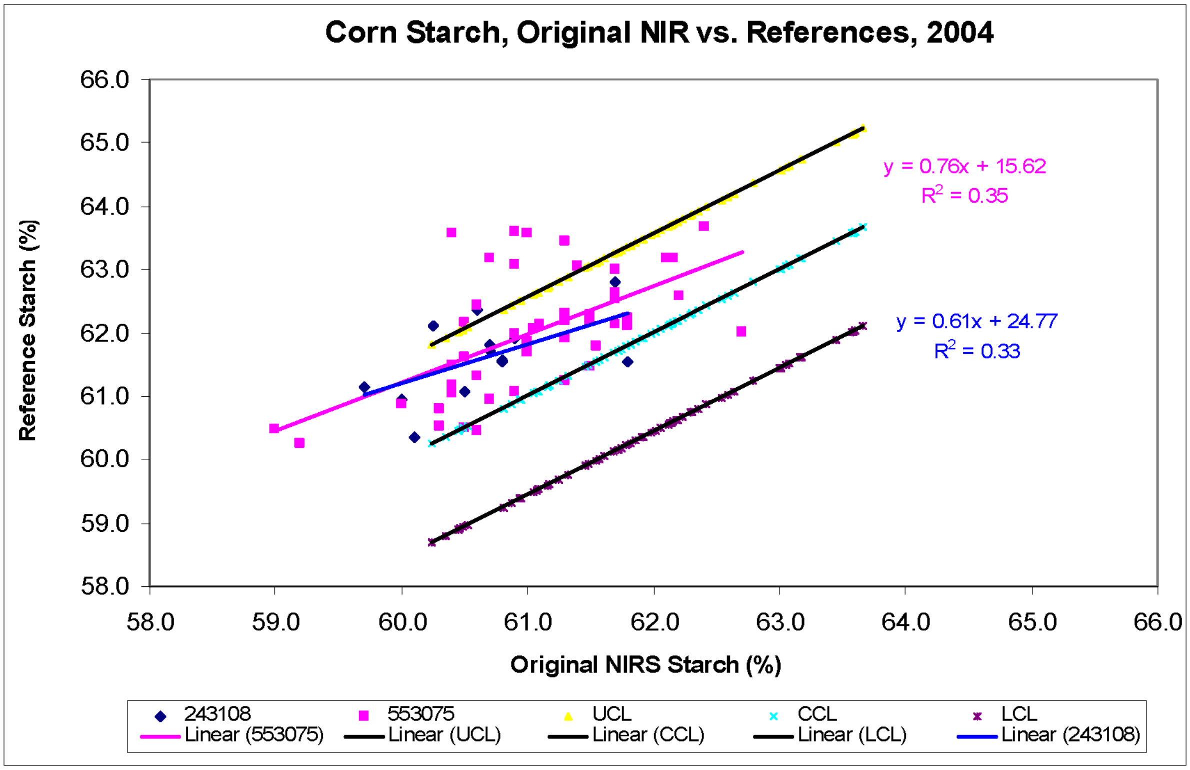


Figure 5. Corn Starch, Original NIRS vs. References, 2004

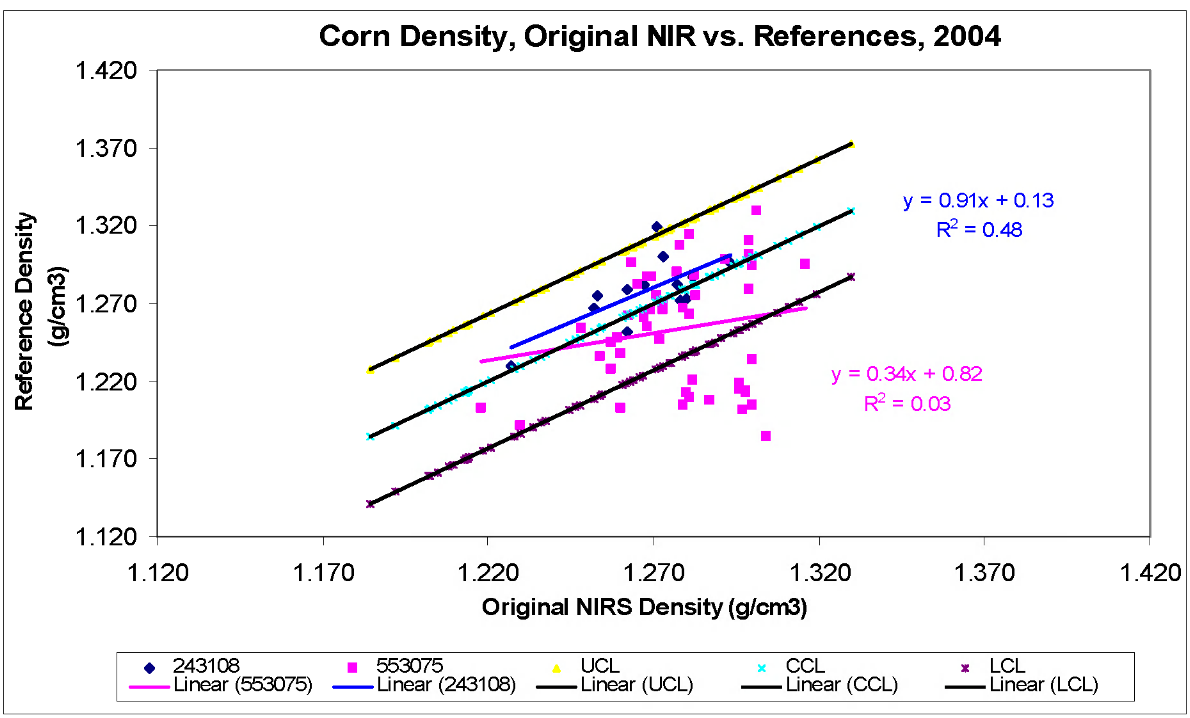


Figure 6. Corn Density, Original NIRS vs. References, 2004

APPENDIX E. SUPPORTING EQUIPMENT

Table 1. Balances Yearly Check, 2004

Date	Balance	No	10 mg	20 mg	50 mg	100 mg	200 mg	500 mg	1 g	2 g	5 g	10 g	20 g	50 g	100 g	250 g	500 g	1000 g
08/25/04	**Denver**	1	0.0100	0.2010	0.0500	0.1001	0.2002	0.4998	1.0004	2.0003	5.0003	10.0002	19.9998	49.9993	99.9976	249.9940		
	A-250	2	0.0099	0.0199	0.0499	0.1006	0.2003	0.5003	1.0004	2.0003	5.0003	10.0002	19.9999	49.9997	99.9978	249.9937		
		3	0.0101	0.0199	0.0504	0.1003	0.2004	0.5001	1.0003	2.0001	5.0002	10.0001	20.0001	49.9993	99.9973	249.9939		
		Ave	0.0100	0.0803	0.0501	0.1003	0.2003	0.5001	1.0004	2.0002	5.0003	10.0002	19.9999	49.9994	99.9976	249.9939		
08/25/04	**AND**	1	0.0099	0.0201	0.0504	0.1004	0.2004	0.5002	1.0008	2.0013	5.0027	10.0046	20.0093	50.0219				
	HR-60	2	0.0098	0.0199	0.0504	0.1002	0.2003	0.5001	1.0008	2.0011	5.0027	10.0051	20.0090	50.0219				
		3	0.0101	0.0200	0.0500	0.1003	0.2003	0.5003	1.0007	2.0010	5.0025	10.0050	20.0091	50.0220				
		Ave	0.0099	0.0200	0.0503	0.1003	0.2003	0.5002	1.0008	2.0011	5.0026	10.0049	20.0091	50.0219				
08/25/04	**Mettler**	1	0.004	0.022	0.056	0.108	0.205	0.500	0.998	1.996	4.996	10.001	20.009	49.987	99.976			
	PB	2	0.017	0.020	0.050	0.104	0.199	0.507	1.000	2.003	5.003	9.995	19.997	49.998	99.970			
	153-5	3	0.016	0.020	0.052	0.104	0.219	0.510	1.001	2.005	5.007	9.994	19.990	49.974	100.023			
		Ave	0.012	0.021	0.053	0.105	0.208	0.506	1.000	2.001	5.002	9.997	19.999	49.986	99.990			
08/25/04	**Mettler**	1	0.00	0.04	0.06	0.10	0.20	0.49	0.99	2.00	4.99	10.00	20.00	50.00	100.01	250.02	499.92	1000.10
	PM	2	0.01	0.02	0.04	0.09	0.21	0.50	1.00	2.00	5.00	10.00	20.00	50.00	100.01	250.02	499.93	1000.08
	4400	3	0.00	0.00	0.06	0.10	0.20	0.51	1.00	2.00	5.00	10.00	20.00	50.00	100.00	250.02	499.93	1000.09
		Ave	0.00	0.02	0.05	0.10	0.20	0.50	1.00	2.00	5.00	10.00	20.00	50.00	100.01	250.02	499.93	1000.09
08/25/04	**Mettler**	1							1	2	5	10	20	50	100	250	500	999
	Toledo	2							1	2	5	10	20	50	100	250	499	1000
	SB	3							1	2	5	10	20	50	100	250	499	999
	16000	Ave							1.0	2.0	5.0	10.0	20.0	50.0	100.0	250.0	499.3	999.3
08/25/04	**Seed**	1					0.0	0.5	1.0	2.0	5.0	10.0	20.0	50.0	100.0	249.9	499.9	1000.2
	Buro	2					0.2	0.5	1.0	2.0	5.0	10.0	20.0	50.1	100.0	249.9	499.9	1000.2
	8800	3					0.2	0.5	1.1	2.0	5.0	9.9	20.0	50.1	100.0	250.0	500.0	1000.3
		Ave					0.1	0.5	1.0	2.0	5.0	10.0	20.0	50.1	100.0	249.9	499.9	1000.2
09/27/04	**Mettler**	1	0.0099	0.0199	0.0500	0.1000	0.2001	0.4997	1.0002	2.0001	5.0001	9.9999	19.9998	49.9992	99.8854			
	AJ	2	0.0099	0.0198	0.0499	0.0999	0.2000	0.4997	1.0001	2.0001	5.0000	10.0000	19.9998	49.9994	99.8855			
	100	3	0.0099	0.0198	0.0499	0.0999	0.2001	0.4997	1.0002	2.0000	5.0001	10.0000	20.0000	49.9993	99.8852			
		Ave	0.0099	0.0198	0.0499	0.0999	0.2001	0.4997	1.0002	2.0001	5.0001	10.0000	19.9999	49.9993	99.8854			
09/27/04	**Mettler**	1	0.00	0.00	0.05	0.10	0.20	0.50	1.00	2.00	5.00	10.00	19.99	49.95	99.79	249.77	499.40	999.00
	PC	2	0.00	0.00	0.04	0.10	0.20	0.50	1.00	2.00	5.00	10.00	19.99	49.96	99.80	249.78	499.40	999.00
	4400	3	0.00	0.00	0.04	0.10	0.20	0.49	0.99	2.00	4.99	9.99	19.99	49.96	99.79	249.77	499.40	999.00
		Ave	0.00	0.00	0.04	0.10	0.20	0.50	1.00	2.00	5.00	10.00	19.99	49.96	99.79	249.77	499.40	999.00

Table 2. Balances Yearly Check, 2005

Date	Balance	No	10 mg	20 mg	50 mg	100 mg	200 mg	500 mg	1 g	2 g	5 g	10 g	20 g	50 g	100 g	250 g	500 g	1000 g
09/06/05	**Denver**	1	0.0101	0.0199	0.0500	0.1001	0.2003	0.4998	1.0003	2.0001	5.0004	10.0007	20.0008	50.0017	100.0022	250.0051		
	A-250	2	0.0098	0.0201	0.0500	0.0997	0.2002	0.4997	1.0002	2.0001	5.0001	10.0005	20.0005	50.0014	100.0021	250.0050		
		3	0.0098	0.0203	0.0500	0.1001	0.2003	0.4998	1.0004	2.0002	5.0003	10.0005	20.0004	50.0012	100.0018	250.0050		
		Ave	0.0099	0.0201	0.0500	0.1000	0.2003	0.4998	1.0003	2.0001	5.0003	10.0006	20.0006	50.0014	100.0020	250.0050		
09/06/05	**AND**	1	0.0098	0.0199	0.0501	0.1000	0.2000	0.4998	1.0002	2.0002	5.0002	10.0003	20.0005	50.0008				
	HR-60	2	0.0100	0.0202	0.0501	0.1001	0.2001	0.4999	1.0002	2.0001	5.0002	10.0004	20.0004	50.0008				
		3	0.0099	0.0203	0.0500	0.1000	0.2002	0.4997	1.0001	2.0001	5.0001	10.0003	20.0004	50.0005				
		Ave	0.0099	0.0201	0.0501	0.1000	0.2001	0.4998	1.0002	2.0001	5.0002	10.0003	20.0004	50.0007				
09/06/05	**Mettler**	1							1.0	2.0	5.0	10.0	20.0	50.0	100.0	250.0	499.0	999.0
	SB	2							1.0	2.0	5.0	10.0	20.0	50.0	100.0	250.0	500.0	999.0
	16000	3							1.0	2.0	5.0	10.0	20.0	50.0	100.0	250.0	500.0	999.0
		Ave							1.0	2.0	5.0	10.0	20.0	50.0	100.0	250.0	499.7	999.0
09/06/05	**Seed**	1					0.20	0.60	1.00	2.00	4.90	10.00	20.00	50.00	99.80	249.90	499.80	1000.30
	Buro	2					0.30	0.40	0.90	2.10	5.00	9.90	19.90	49.90	100.00	249.90	499.90	1000.30
	8800	3					0.20	0.50	1.10	2.10	5.00	10.00	20.00	50.10	99.90	249.80	499.80	1000.20
		Ave					0.23	0.50	1.00	2.07	4.97	9.97	19.97	50.00	99.90	249.87	499.83	1000.27
09/06/05	**Mettler**	1	0.010	0.020	0.050	0.100	0.199	0.500	1.000	2.001	5.001	10.000	20.001	50.001	99.889			
	PB	2	0.010	0.019	0.050	0.099	0.199	0.500	1.000	2.001	5.000	10.000	19.999	50.000	99.889			
	153-S	3	0.011	0.020	0.049	0.100	0.200	0.500	0.999	2.000	5.001	10.000	20.000	50.001	99.888			
		Ave	0.010	0.020	0.050	0.100	0.199	0.500	0.9997	2.001	5.001	10.000	20.000	50.001	99.889			
09/06/05	**Mettler**	1	0.0097	0.0201	0.0503	0.1002	0.2003	0.4999	1.0003	2.0001	5.0001	10.0005	20.0008	50.0011	99.889			
	AJ	2	0.0099	0.0201	0.0499	0.0998	0.2000	0.4997	1.0001	2.0002	5.0003	10.0008	20.0011	50.0014	99.888			
	100	3	0.01	0.02	0.0502	0.0999	0.2002	0.4998	1.0003	2.0003	5.0002	10.0006	20.0007	50.0012	99.889			
		Ave	0.010	0.020	0.050	0.1000	0.2002	0.4998	1.0002	2.0002	5.0002	10.0006	20.0009	50.0012	99.889			
09/06/05	**Mettler**	1			0.05	0.10	0.20	0.50	1.00	2.00	5.00	10.00	20.00	50.00	99.89	249.89	499.90	1000.05
	PM	2			0.04	0.10	0.19	0.50	1.00	2.00	5.00	10.00	20.00	50.00	99.89	249.89	499.90	1000.04
	4000	3			0.05	0.10	0.20	0.50	1.00	2.01	5.01	10.00	20.00	50.00	99.89	249.90	499.90	1000.04
		Ave			0.05	0.10	0.20	0.50	1.00	2.00	5.00	10.00	20.00	50.00	99.89	249.89	499.90	1000.04

APPENDIX F. REPLICATION NUMBER BEFORE AND AFTER OUTLIER REMOVAL (REFERENCE DATA)

Table 1. Number Measurements of Individual Soybeans Sample Before and After Outlier Removal

No	Sample Number	Number of Reps (Oil)		Number of Reps (Protein)	
		Before	After	Before	After
1	19960299	15	10	14	10
2	19970172	9	7	8	5
3	19980002	13	10	12	9
4	19991674	9	6	8	6
5	19991675	8	5	7	6
6	20000771	8	5	7	5
7	20020199	8	6	7	5
8	20020226	9	7	8	5
9	20020416	9	8	8	6
10	20020570	8	5	8	5
11	20020572	8	6	6	3
12	20030026	10	7	9	7
13	20040358	3	2	3	2
14	20040369	4	3	4	3
15	20040370	4	3	4	3
16	20040388	3	2	3	2
17	20040493	4	3	4	2
18	20040607	4	3	4	3
19	20040609	4	3	4	3
20	20040682	3	2	3	2
21	19980001	14	10	11	8
22	19991671	9	6	9	5
23	20010461	6	4	5	4
24	20020201	7	5	6	4
25	20020567	6	4	5	4
26	20030028	6	4	5	3
27	20040608	2	2	2	2
28	20040598	4	3	4	2
29	20040602	2	2	2	2
30	20000001	6	4	5	4
	Total	205	147	185	130

Table 2. Number Measurements of Individual Corn Sample Before and After Outlier Removal

No	Sample Number	Number of Reps (Oil)		Number of Reps (Protein)		Number of Reps (Density)	
		Before	After	Before	After	Before	After
1	19960736	9	6	9	9	6	6
2	19960763	6	4	6	6	8	8
3	19960764	9	5	8	6	6	4
4	19960765	5	3	5	3	4	3
5	19970018	5	3	5	4	6	4
6	19980119	8	6	8	5	5	3
7	19990958	7	5	7	6	3	2
8	19990960	6	4	7	5	3	2
9	19990998	5	3	6	4	2	2
10	19990999	7	4	6	4	3	2
11	20000891	5	3	3	2	2	2
12	20010673	6	3	3	2	4	3
13	20010687	4	2	4	3	2	2
14	20010688	3	2	3	2	2	2
15	20010717	4	3	4	3	3	2
16	20010718	4	3	4	3	2	2
17	20020020	3	2	3	2		
18	20020352	3	2	4	3	2	2
19	20020353	3	2	3	2	2	2
20	20020355	5	3	3	2	2	2
21	20020357	3	2	3	2	2	2
22	20020359	6	3	4	2	2	2
23	20030027	3	2	3	2		
24	20030151	3	2	3	2	2	2
25	20030455	3	2	3	2	2	2
26	20030457	3	2	3	2	2	2
27	20030459	3	2	3	2	2	2
28	20040154	3	2	3	2		
29	20040263	3	2	3	2		
30	20040327	2	2	2	2		
31	19960761	5	3	5	4		
32	19970020	5	3	4	3	7	4
33	19990997	7	5	7	6	5	3
34	19991000	8	6	6	5	4	3
35	20010672	4	3	4	2	2	2
36	20010719	4	2	4	2	2	2
37	20020348	5	4	3	2		
38	20020358	4	3	4	2		
39	20030153	3	2	3	2		
	Total	184	120	171	124	99	81

APPENDIX G. AMINO ACID TOLERANCE LIMITS

Table 1. Soybeans Amino Acid Tolerance Limits

Amino Acid	Standard Deviation (%)	CV	LCL (%)	UCL (%)
Taurine	0.03	71.06%	-0.07	0.07
Hydroxyproline	0.02	42.52%	-0.06	0.06
Aspartic Acid	0.07	1.66%	-0.19	0.19
Threonine	0.02	1.37%	-0.05	0.05
Serine	0.09	5.61%	-0.26	0.26
Glutamic Acid	0.21	3.23%	-0.59	0.59
Proline	0.05	3.10%	-0.15	0.15
Lanthionine	0.03	91.95%	-0.07	0.07
Glycine	0.03	1.87%	-0.08	0.08
Alanine	0.03	1.90%	-0.08	0.08
Cysteine	0.03	4.52%	-0.08	0.08
Valine	0.06	3.24%	-0.16	0.16
Methionine	0.02	4.08%	-0.06	0.06
Isoleucine	0.05	2.75%	-0.13	0.13
Leucine	0.06	2.10%	-0.17	0.17
Tyrosine	0.03	2.45%	-0.09	0.09
Phenylalanine	0.03	1.80%	-0.09	0.09
Hydroxylysine	0.01	147.58%	-0.02	0.02
Histidine	0.03	3.26%	-0.09	0.09
Ornithine	0.01	69.31%	-0.04	0.04
Lysine	0.04	1.59%	-0.10	0.10
Arginine	0.03	1.25%	-0.09	0.09
Tryptophan	0.07	15.96%	-0.19	0.19
Total Sulfur AA	0.04	3.98%	-0.12	0.12
5 Key AA	0.11	2.12%	-0.31	0.31
Total AA	0.47	1.36%	-1.32	1.32

Table 2. Corn Amino Acid Tolerance Limits

Amino Acid	Std.dev (%)	CV	LCL (%)	UCL (%)
Taurine	0.07	85.90%	-0.20	0.20
Hydroxyproline	0.01	43.89%	-0.02	0.02
Aspartic Acid	0.03	5.61%	-0.08	0.08
Threonine	0.01	5.43%	-0.04	0.04
Serine	0.02	7.05%	-0.06	0.06
Glutamic Acid	0.05	3.98%	-0.15	0.15
Proline	0.02	3.33%	-0.06	0.06
Lanthionine	0.00	135.88%	-0.01	0.01
Glycine	0.01	4.69%	-0.04	0.04
Alanine	0.02	4.05%	-0.06	0.06
Cysteine	0.01	6.09%	-0.03	0.03
Valine	0.02	4.75%	-0.05	0.05
Methionine	0.02	9.06%	-0.04	0.04
Isoleucine	0.01	5.34%	-0.04	0.04
Leucine	0.03	3.70%	-0.09	0.09
Tyrosine	0.01	4.87%	-0.03	0.03
Phenylalanine	0.01	4.03%	-0.04	0.04
Hydroxylysine	0.00		0.00	0.00
Histidine	0.02	9.71%	-0.05	0.05
Ornithine	0.01	104.09%	-0.02	0.02
Lysine	0.02	7.35%	-0.05	0.05
Arginine	0.02	6.04%	-0.06	0.06
Tryptophan	0.01	22.71%	-0.03	0.03
Total Sulfur AA	0.02	6.24%	-0.06	0.06
5 Keys AA	0.05	5.07%	-0.13	0.13
Total AA	0.30	4.05%	-0.82	0.82

APPENDIX I. OVEN MOISTURE

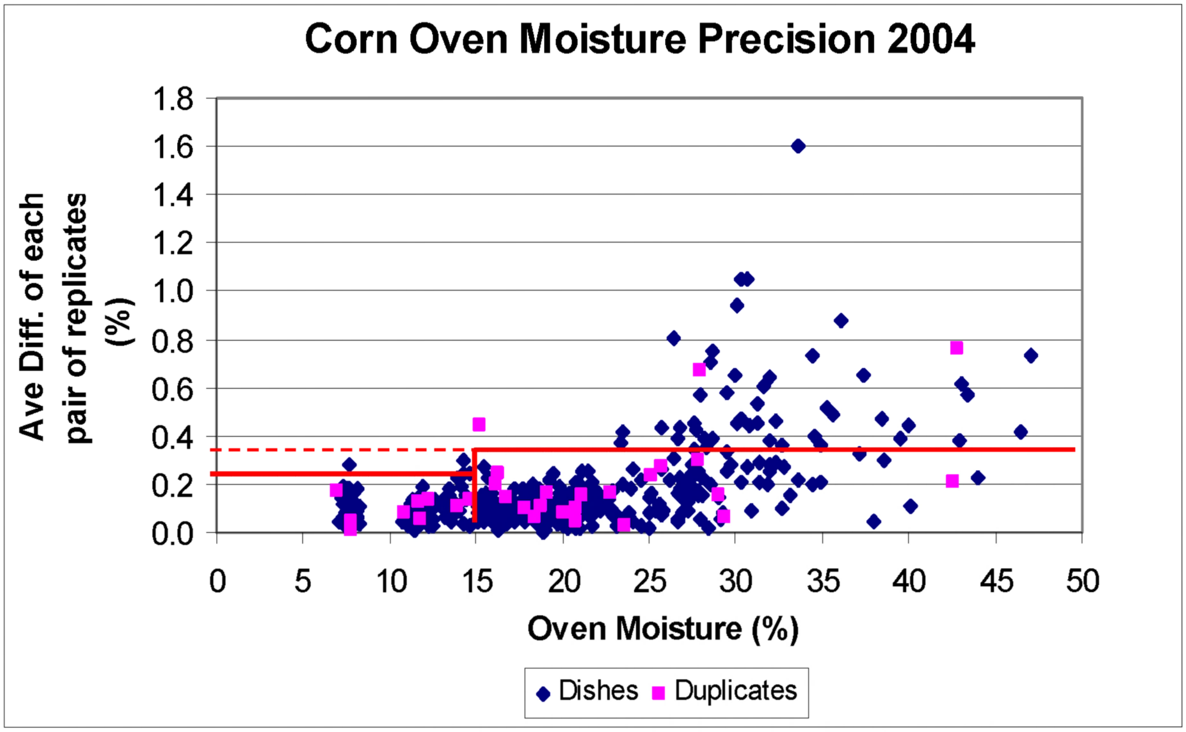


Figure 1. Corn Oven Moisture Precision Chart, 2004

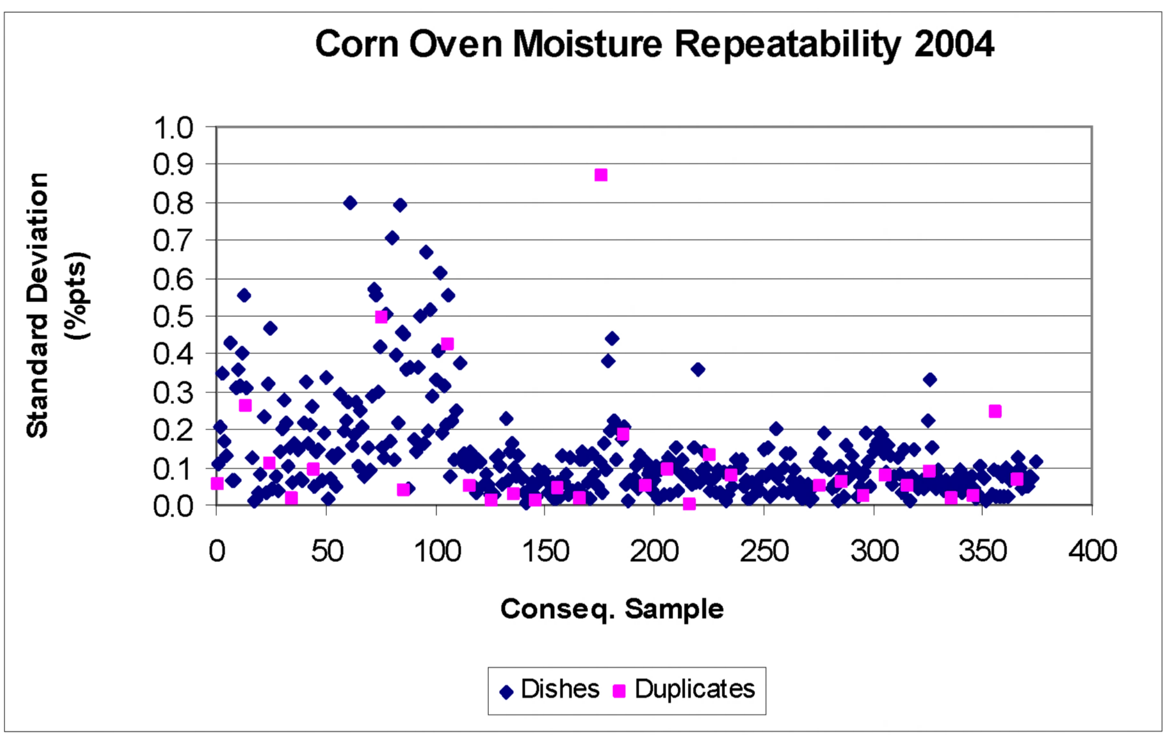


Figure 2. Corn Oven Moisture Repeatability Chart, 2004

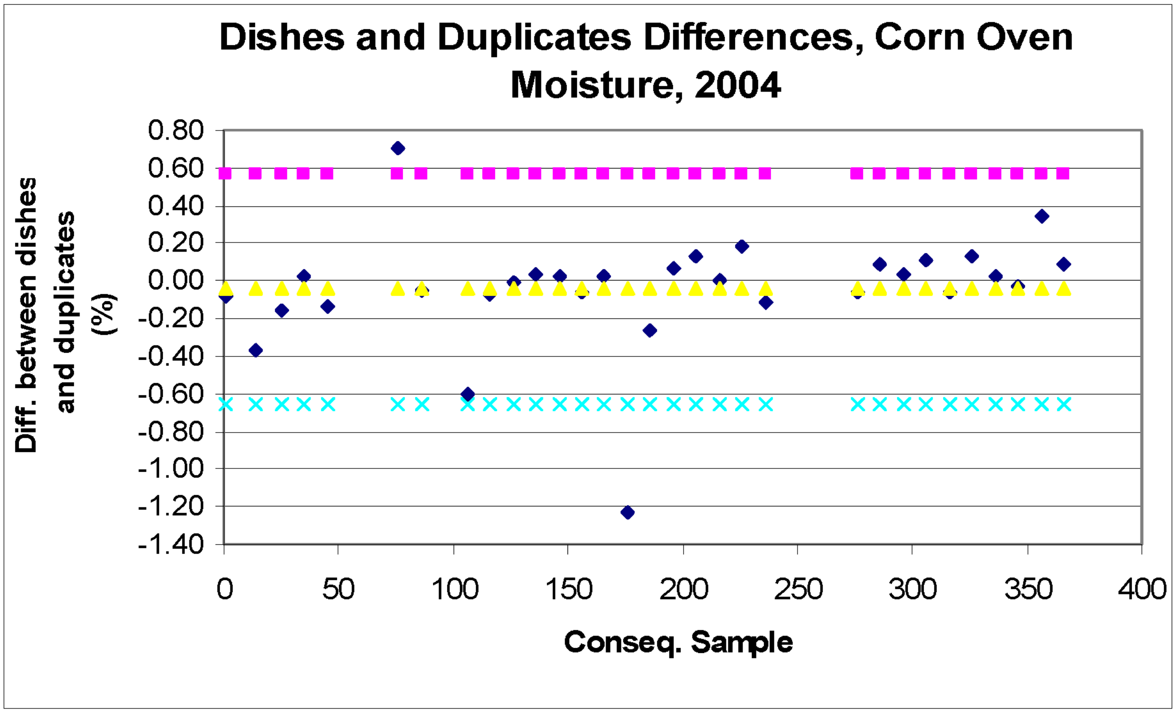


Figure 3. Differences between Dishes and Duplicate of Corn Oven Moisture Chart, 2004

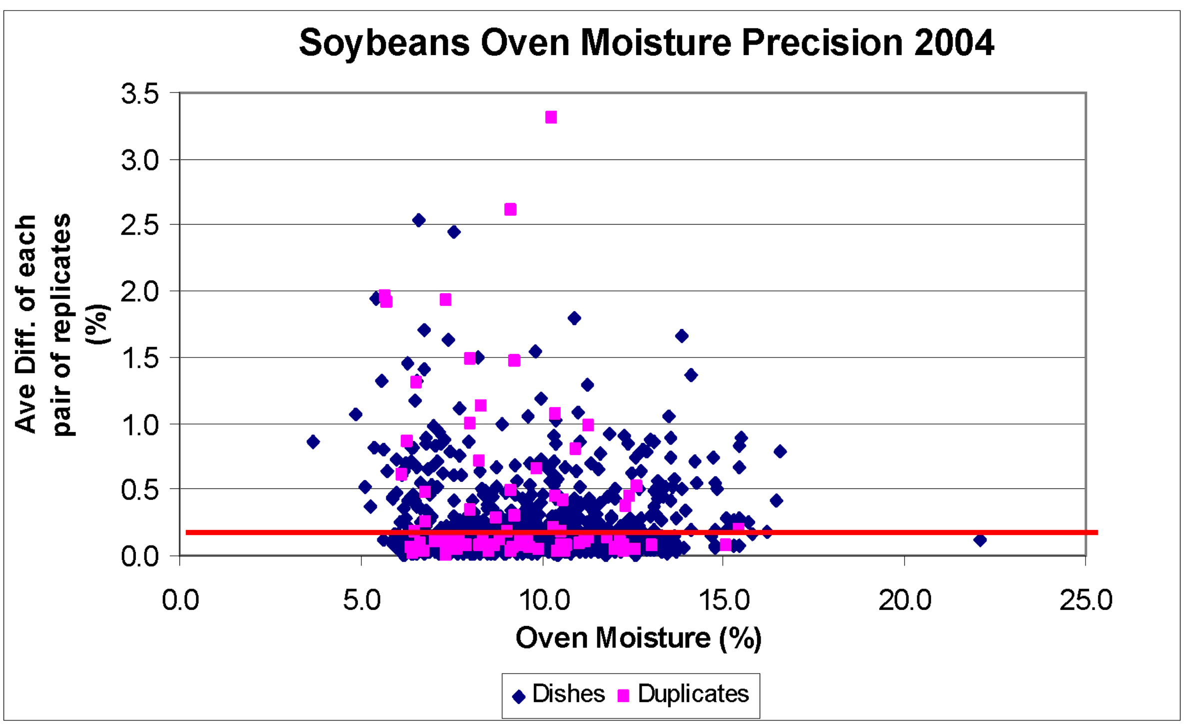


Figure 4. Soybeans Oven Moisture Precision Chart, 2004

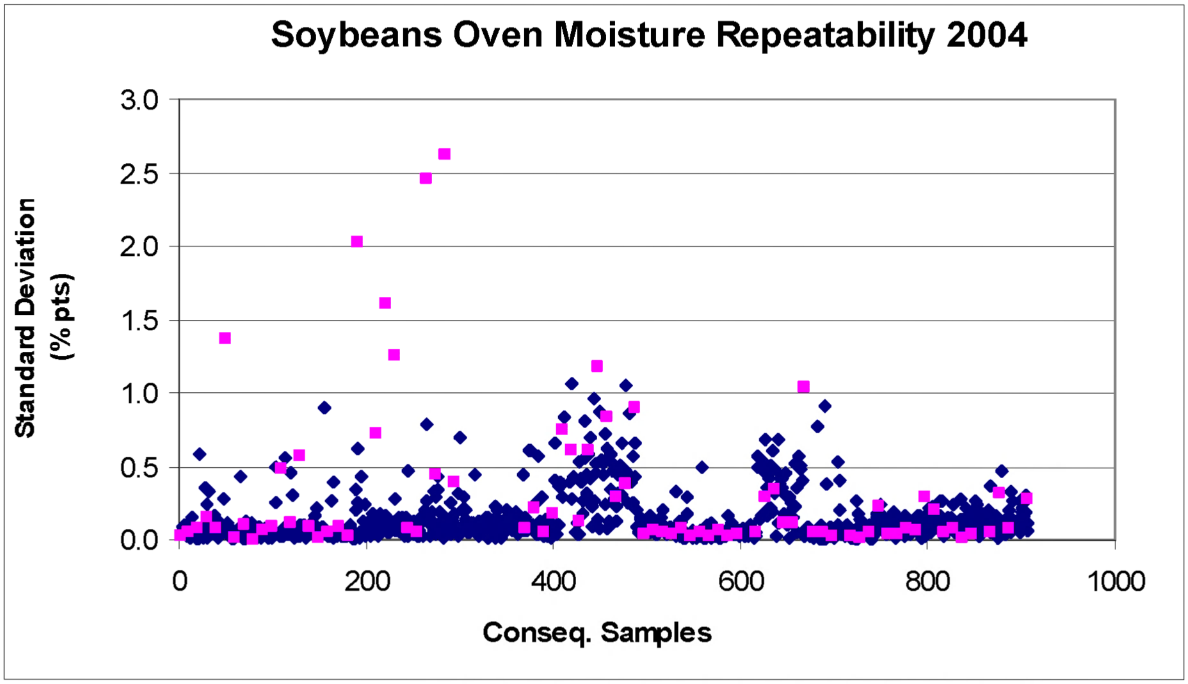


Figure 5. Soybeans Oven Moisture Repeatability Quality Control Chart, 2004

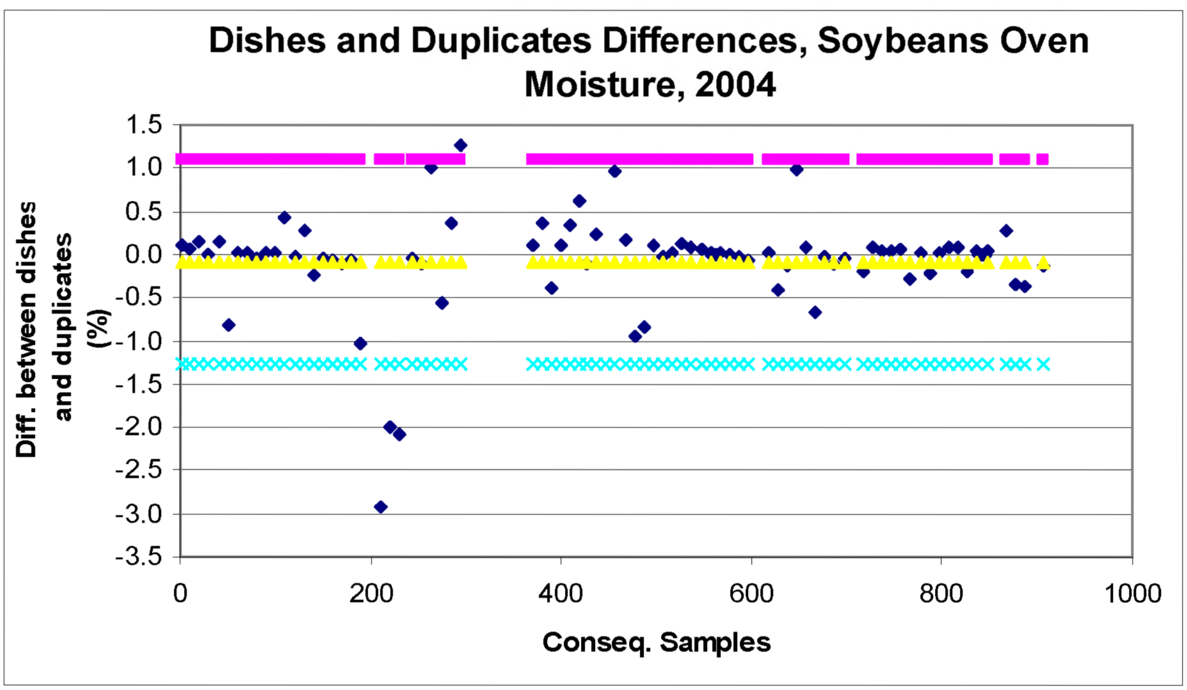


Figure 6. Differences between Dishes and Duplicate of Soybeans Oven Moisture Chart, 2004

APPENDIX I. QUALITY CONTROL ACTIVITY PROCEDURES

Grain Quality Laboratory
Iowa State University

PROCEDURE: NIRS Daily Check Sample

Background:
ISU Grain Quality Lab has many instruments that are used to calibrate grain samples from many areas in United States. In order to maintain the performance of the instruments, sample check is done daily in those instruments. Daily sample check is also completed as a part of research in this laboratory.

Objective:
- To maintain the performance of the instruments.
- To make sure that the instruments are stable.
- To collect data as a part of NIRS research.

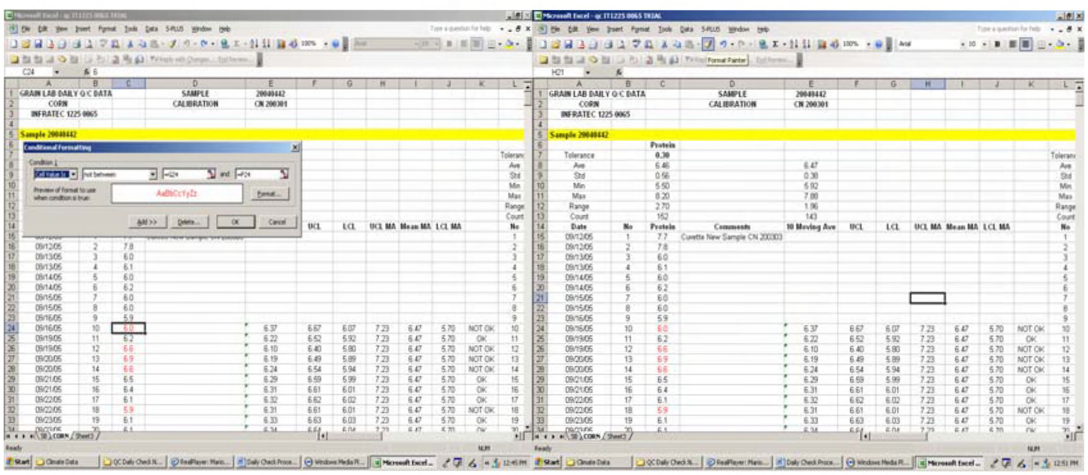
OPERATION

Procedure Detail:
1. Turn on every NIRS instrument in the lab, let them to warm up.
2. Ask the lab manager for the password.
3. Set up all the instruments so that they are ready to be used for daily sample check.
4. Check the temperature of the sample (corn and soybeans) using the IR thermometer.
5. Record the today's date and sample temperatures on the NIRS books. (Use yellow sheet for corn and green sheet for soybeans, the sheets are available in the lab (in the one of the shelf).
6. Follow the procedure of every instrument.
7. Choose the right name and type of the sample (corn or soybeans)
8. Choose the right calibration ID.
9. Enter the sample number, temperature and date.
10. Put the sample in the place that each instrument has provided.
11. Hit confirm, start sample scan or measure button.
12. Record the results on the NIRS books.
13. Do the test two times on corn and soybeans.

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:
1. Do the check sample on every NIRS instrument daily.

2. Do the test two times on corn and soybeans.
3. Record the date of test, sample temperature and test results on every book of NIR instrument.
4. Record the daily check data (corn: protein, oil, starch, density; soybeans: protein and oil) into spreadsheet weekly. The spreadsheets are available at \\Grainbin\QCdata\QC daily checks new tol. Every NIR instrument has its own spreadsheet.
5. Update the 10-point moving average, UCL MA, LCL MA, UCL, Mean and MA by copying the formula from previous data/measurement.
6. Format the cell of the data(corn: protein, oil, starch, density; soybeans: protein and oil) using Format-conditional formatting, choose cell value is **not between UCL MA and LCL MA cell number** (This UCL and LCL have to be the same as data row's number), click format, change the format color to red**. Use the format painter to copy the format to the next rows (**Click on the data that have had format, then drag the painter to the next rows).



7. The out of control data will be turned red.
8. Update the condition by copying from previous row, to double check whether the data is "OK" or "NOT OK".
9. Update the control chart by dragging the data, UCL MA, LCL MA, by. Sometimes the chart can update automatically if all the series has been dragged to the many empty lines before.
10. When the sample is changed:
    - Update the last line of average, standard deviation, min, max, range and count of old samples.
    - Update the last line in the control charts, make sure they are right.
    - Type the new sample number and highlight it using yellow color.
    - Copy the format and formula from the previous sample.

- Build the new control charts by copying from previous sample and change their start line/row.

11. Record the activities happen to the instrument in the log book of instrument and in the spreadsheet (comments column).
12. Print the updated chart and put in the LAB QC book (monthly).
13. Make the quarterly report, the format of report is available at \\Grainbin\QCdata\Equipment QC\Daily Check Report.

THE USE OF DATA

The NIR daily check data are collected for the quality control purposes. It is done to check the precision and stabilization of every NIRS instrument. The errors can be detected by analyzing the daily check data. The action can be determined and done before the instruments are used to analyzed samples (for the services and others activities). By doing quality control in every instrument, Grain Quality Lab will be able make sure that the results are reliable.

Control Chart Guidelines:

- When there is point that suddenly fall far away from the others points:
  - Check the data if there is typo (check the original data in the instrument QC book).
  - If the original data is the same with the data entered into spreadsheet, check the original data that are saved in the instrument, there may be error recording by operator.
- When the out of control data is more than 5%, there are some possibility:
  - Instrument has problem.
  - Check sample need to be replaced.
  - Operator training is needed.
- The exact causes of unstable instrument may be searched using root cause analysis and the problems should be discussed during weekly staff meeting.

Evaluation and Monitoring:

- Do the check sample on every NIRS instrument daily.
- Analyze the data and chart weekly after entering data.
- Print the control charts monthly and place it in the QC book of every instrument.
- Update the report quarterly.
- The data should be between LCL and UCL
- The out of control data should not exceed 5% of the overall data.

Corrective Actions:

- Redo the test when the result is unusual compare to the previous results.
- Make sure that the test is done using correct procedure.

- When the out of control data exceed 5% of the overall data, follow the guidelines above.
- Consult with the Lab manager or discuss the problem during weekly staff meeting.

Grain Quality Laboratory
Iowa State University

PROCEDURE: NIRS Instrument Duplicates

Background:
The NIRS instruments especially the instruments that are used for the services need to be checked for their precision and reproducibility. The ten percent of measurements using NIRS instruments are duplicated to measure the reproducibility over time

Objective:
To measure the reproducibility over time of the NIRS instruments used for the services.

OPERATION AND DATA COLLECTION

1. Open the standard service template found in
   \\Grainbin\Service\200X\Corn\CNDATAFORM0X.xls for corn -or-
   \\Grainbin\Service\200X\Soybeans\sbDATAFORM0X.xls for soybeans.
   "X" is the last digit of the year.
2. "Save As" the spreadsheet in the correct folder for corn or soybeans using the following naming convention: \R cn (or sb) project descriptor set number.
3. Complete the date and source of the test for every line of data.
4. Run samples, collect duplicate data at least every tenth sample. Confirm the number of duplicates to be taken with the supervisor. Duplicate data records update automatically on the Dup Collection tab.
5. Save the spreadsheet usually after every sample.

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:

1. Open the duplicate summary from the previous year at \\Grainbin\QCdata\Duplicate Summary\XXX, copy them to the new file, save as **Duplicate Corn** or **Duplicate Soybeans**, put them to the new folder, name the folder using the respective year.
2. Delete the data except the first data to keep the formula.
3. Collect the duplicate data from service file at :
   \\Grainbin\Service\XXXX\Corn
   \\Grainbin\Service\XXXX\Strip Plots\Corn
   \\Grainbin\Service\XXXX\Strip Plots\Soybeans
   \\Grainbin\Service\XXXX\Soybeans

4. Make a new sheet on the service file "Duplicate", copy all the data to the sheet, delete the data without duplicates.
5. Copy the first and second results to the duplicate summary file according to the instrument that is used, follow the format.
6. Record the service file name in first column to make sure no multiple records.
7. Records the service file name in the sheet of paper so next time you don't need to reopen files that have been recorded.
8. Copy the format/formula from the first data (from previous file)
9. Delete the first data from the previous file.
10. Update the duplicate different control chart, change the file and sheet name in the control chart (match them with the file), make sure they are not from previous file data.
11. Do data collection continuously; do not wait until the services are done for the certain year.
12. Post the updated control chart to the respective NIRS instrument book.
13. When the duplicate data collection has done, open the QC Duplicate Summary file at \\Grainbin\QCdata\Duplicate Summary\QC Duplicate Summary Summarize the average, standard deviation, average of differences. , standard deviation of differences and number of data (n) every instrument into table, follow the format from previous year. Update the summary of every instrument table and different between year tables, follow the format.
14. Do quarterly report; follow the format at \\Grainbin\QCdata\Duplicate Summary\Duplicate Instrument Report.

THE USE OF DATA

The duplicate data and the control chart are collected for the quality control purposes. This data will be analyzed to determine whether the NIRS instrument has acceptable reproducibility level. The errors can be prevented before the activities go further.

Control Chart Guidelines:

- The points should be random variation and not form a trend.
- When the data form a trend, there may be error with the instrument.
- When there is point that jumps from other points:
  - There may be error with the instrument
  - Operator does error, for example: the result is actually the result of the next sample.
- When the out of control data exceed 5%, it means that the instrument reproducibility is not good. The exact causes should be searched using root cause analysis. The problems also need to be discussed in the weekly staff meeting.

Evaluation and Monitoring:

- Collect the duplicate data when lab does the services to customers.
- Process the data as soon as possible (the data are processed until the service activity during certain year finish).
- Print the control charts monthly.
- Analyze the data and control chart.
- Update the report quarterly.
- The out of control data should not exceed 5% of the total data.

Corrective Actions:

- When the differences between first and second run, you will be notified to rerun the sample.
- When you forget to measure the tenth sample twice, do the duplication using next sample.
- If the out of control data exceed 5% of the total data, consult with the lab manager or discuss the problem in the weekly staff meeting to determine what action should be taken.

Grain Quality Laboratory
Iowa State University

PROCEDURE: Original NIRS vs. References

Background:
The original NIRS values are obtained when a calibration sample run for the first time, before it is known that it is a calibration sample. In order to check the accuracy of NIRS instrument, these values then are compared with the reference values from the chemistry lab.

Objective:
To check the accuracy of the NIRS instrument with the respect of reference chemistry values and vice versa.

Procedure Detail:
1. Open the calibration file at \\Grainbin\Nircal\CNCALXX for corn or \\Grainbin\Nircal\SBCALXX for soybeans. The calibration files are named according to the year.
   For example: CNCAL2004.
2. Copy the "overall" sheet into new file, save as Original NIR vs. References XXXX in the \\Grainbin\QCdata\Original NIR vs. References new.
3. Keep the column: ID1(customer), ID3, Universal ID, Origin, Protein, Oil , Starch and Density at 15% basis moisture for corn and Protein and oil at 13% basis moisture.
4. Delete the others columns.
5. Delete the samples that do not have chemistry values or only has moisture values.
6. Open the service file at \\Grainbin\Service\XXXX\Corn/, \\Grainbin\Service\XXXX\Strip plots/Corn by looking at "original" column. Some samples might be located at \\Grainbin\Surveys\Soybeans (usually soybeans sample).
7. Match the universal ID in the calibration file with the ID in the service file.
8. Copy the Protein Oil, Starch, Density values for corn and Protein, Oil values for soybeans.
9. Record the instrument that is used for the services.
10. After done with file searching and copying, make some new columns according to the instruments serial number like the example below:

Moisture			Protein			Oil		
553792	553075	65	553792	553075	65	553792	553075	65

11. Rearrange the results according to their instruments.

12. Build the chart for every chemistry item (Corn: protein, oil, starch, density; Soybeans: protein, oil) X axis is the original NIRS values and Y axis is the references chemistry values. Make series with different colors according to the instrument serial numbers.
13. Add trend line (linear line) to the graph for every series/instrument.
14. Make new columns for the UCL, CCL and LCL for every item (Corn: protein, oil, starch, density; Soybeans: protein, oil). CCL= Original NIRS (This will form y=x line), UCL /LCL = Original NIR values ± Tolerance (95% CI) (This will form y= x ± tolerance)

(you can check the tolerance values from previous year files)

THE USE OF DATA

The comparison of original NIRS data and data from chemistry lab are created for the quality control purposes. These data are used to measure the accuracy of NIRS instrument against chemistry lab.

Chart Guidelines:

- The regression line of original NIRS data against chemistry lab data should be close to target line (y=x line). The r-square should be close to 1.
- If the regression line is not close to target line (r-square is really far from 1), there may be problem with instrument or chemistry data. Check the actual calibration data; whether they show a good result (r-square is close to 1).
- Discuss the problems during weekly staff meeting.

Evaluation and Monitoring:

- Analyze the correlation between Original NIRS and reference chemistry data
- Analyze data that are not in control.
- Print the charts quarterly.
- Update the report yearly.

Corrective Actions:

- Redo the measurement using NIRS instrument for the samples that have a big difference between the reference chemistry and NIRS
- Compare the results with the reference chemistry values.
- If the difference is still big, send those samples chemistry to the chemistry lab.
- Compare the results again to determine where the problems are located.

Grain Quality Laboratory
Iowa State University

PROCEDURE: Balance Checks

Background:
Balances are used often to find the weight of the samples. If the balances are inaccurate the data will not be accurate.

Objective:
To check if the balances are working accurately.

OPERATION

Procedure Detail:

15. Get the various weights from the weights drawer in room 1546 and the balance check sheets. You will need a box containing weights from 10mg to 50g. This box contains a pair of tweezers for handling the smaller weights (10mg to 500 mg). NEVER USE YOUR HANDS TO HANDLE THESE WEIGHTS. You will also need five – 100g weights and one 1000g weight.
16. This procedure is done on seven balances.
    i. Denver Instrument A- 250
    ii. AND HR-60
    iii. Mettler AJ100
    iv. Mettler Toledo PB153-S
    v. Mettler PM4400
    vi. Mettler Toledo SB16000
    vii. Seedburo 8800
17. Start weighing from the smallest to the largest. Recording the results on the balance check sheets as they appear on the balance. Check only the weights that are within the balance weight range. Do not exceed the maximum weight range of the balance.
18. To do balance check on <u>Denver Instrument A – 250</u>
    i. This balance has a weight range of 10 mg – 250g.
    ii. Make sure the units are on grams and balance is on zero. If the balance is not on zero press [TARE]
    iii. Put the weight on the balance starting with the smallest weight (10 mg). Letter U will appear next to grams when the machine is balancing. Wait until the letter disappears and the numbers are stable.
    iv. Write down the weight on the balance sheet as it appears on the screen.
    v. Remove the weight and put it back in the box
    vi. Wait until the balance returns to zero. If it doesn't press [TARE]

vii. Put the next weight on the balance and repeat steps (iii – vi)
viii. Continue following the same pattern until you reach the maximum weight of the balance.

19. Repeat the same procedure (no. 4) three times
20. Go the next balance.
21. To do balance checks on AND HR-60
    i. This balance has a weight range of 10 mg – 50g.
    ii. Put the weight on the balance starting with the smallest weight. Wait until the numbers are stable.
    iii. Write down the weight on the balance sheet as it appears on the screen.
    iv. Remove the weight and wait until the balance returns to zero. If the balance is not on zero press [re zero]
    v. Put the next weight on the balance and repeat steps (iii – iv)
    vi. Go to the next weight and repeat the same procedure as above.
22. Repeat the same procedure three times
23. Go the next balance.
24. To do balance checks on Mettler AJ100
    i. This balance has a weight range of 10 mg – 100g.
    ii. If the balance is not on zero press [→O/T←]
    iii. Follow the same procedure as above.
25. To do balance checks on Mettler Toledo PB153-S
    i. This balance has a weight range of 10 mg – 100g.
    ii. If the balance is not on zero press [→O/T←]
    iii. Follow the same procedure as above.
26. To do balance checks on Mettler PC4400
    i. This balance has a weight range of 10 mg – 1000g.
    ii. If the balance is not on zero press the grey tab
    iii. Follow the same procedure as above
13. To do balance checks on Mettler PM4400
    i. This balance has a weight range of 10 mg – 1000g.
    ii. If the balance is not on zero press the grey tab
    iii. Follow the same procedure as above
14. To do balance checks on Mettler Toledo SB16000
    i. This balance has a weight range of 1g – 1000g.
    ii. If the balance is not on zero press [→O/T←]
    iii. Follow the same procedure as above
15. To do balance checks on Seedburo 8800
    1. This balance has a weight range of 200 mg – 1000g.
    2. If the balance is not on zero press [zero]
    3. Follow the same procedure as above

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:

1. Print the balance monthly/yearly check form which is available at \\Grainbin\QCdata\Equipment QC\Balances\monthly check form.
2. Record the date of test on equipment check interval located near the oven moisture.
3. Do the full scale balance check yearly.
4. Do the single point balance check monthly.
5. Follow the operation procedure when doing the test on each balance.
6. Do three times measurement on each balance.
7. Record the date of test and test result in the form and put it in the LAB QC binder.
8. Record the data in the spreadsheet at \\Grainbin\QCdata\Equipment QC\Balances/monthly check.
9. Calculate the deviation percentage by copying formula from previous data/measurement, the formula is already set.
10. Update the chart, by dragging the deviation percentage data for every balance. Sometimes the chart can update automatically if the all the series has been dragged to the many empty lines before.
11. Print the updated chart and put in the LAB QC book.
12. Make the quarterly report, the format of report is available at \\Grainbin\QCdata\Equipment QC\Equipment Check Report.

THE USE OF DATA

The balance check data as a part of quality control procedure will be used for the NIRS calibration purposes. The data from SeedBuro 8000 will affect the accuracy of test weight. The data from Mettler Toledo PB153-S will affect the oven moisture data since this balance is used to weight the sample before going to the oven moisture.

Evaluation and Monitoring:

a. Do quality control and maintenance yearly for full scale and monthly for single point in these balances:
   1. Denver Instrument A- 250: 100g
   2. AND HR-60: 20g
   3. Mettler AJ100: 50g
   4. Mettler Toledo PB153-S: 50g
   5. Mettler PM4000: 50g
   6. Mettler Toledo SB16000: 1000g
   7. Seedburo 8800: 1000g

b. Analyze the data and chart when doing the monthly check.
c. Print the control charts and place it the Lab QC book quarterly.
d. Update the report quarterly.

Corrective Actions:

- Redo the measurement when the difference between current and known measurement are not within the tolerance.
- When the measurement still show that the difference between current and known measurement are not within the tolerance, check the balance and consult with the Lab manager to repair the balance.

Grain Quality Laboratory
Iowa State University

PROCEDURE: Thermometers Check

Background:
Thermometers are used to measure the sample temperature before the samples are tested in the certain instruments. If the thermometers are inaccurate, the data will not be accurate.

Objective:
To check if the thermometers are working accurately.

OPERATION

Procedure Detail:

1. Boil the water to check thermometers in the boiling water temperatures
2. Put and keep the water in refrigerator for one day to check thermometer in refrigerator temperature.
3. Check thermometer in the grain for room temperature.
4. Do step (1-3) three times for both IR and Mercury (Glycol for the new ones) thermometer.

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:

1. Print the thermometer check form which is available at \\Grainbin\QCdata\Equipment\Thermometers.
2. Record the date of test on equipment check interval located near the oven moisture.
3. Do the test on thermometers monthly.
4. Do three times measurement on each condition (boiling water, refrigerator and room).
5. Record the date of test and test results in the form and put in the LAB QC book.
6. Record the data in the spreadsheet at \\Grainbin\QCdata\Equipment\Thermometers
7. Calculate the difference between IR and mercury (or average of three glycol thermometers) data by copying the formula from previous data/measurement.
8. Update the chart by dragging the difference results. Sometimes the chart can update automatically if the all the series has been dragged to the many empty lines before.
9. Print the updated chart and put in the LAB QC book.
10. Make the quarterly report, the format of report is available at \\Grainbin\QCdata\Equipment QC\Equipment Check Report

THE USE OF DATA

The thermometer data as a part of quality control procedure will be used for the NIRS calibration purposes. The IR thermometer data will affect the accuracy of measurements using NIRS instruments. The periodic check is done to make sure the device is ready to use and accurate. Since the NIRS instruments are very sensitive to the temperature, the accuracy of temperature measured is very important.

Evaluation and Monitoring:

- Do Quality Control and maintenance monthly.
- Analyze the data and chart.
- The difference between Mercury or (Glycol) and IR should not exceed the tolerance limitsl limits ($\pm 2^0$C)
- Print the control charts and place it in the Lab QC book.
- The out of control data should not exceed 5% of the total data.

Corrective Actions:

- Redo the measurement when the results are undesirable.
- Replace the battery of IR thermometer when needed.
- Recalibrate the IR thermometer when the differences between the IR and mercury (Glycol) thermometer are out of control.
- Use the instructions for IR thermometer calibration correctly.
- When the out of control data exceed 5% of the total data after all steps above are done, consult with lab manager to do appropriate action (thermometers probably need to be changed).

Grain Quality Laboratory
Iowa State University

PROCEDURE: Boerner Grain Divider

Background:
Sometimes only a part of the sample needed during the lab procedure. The grain divider divides the sample into two equal portions.

Objective:
To equally divide the sample into two portions. (50% of weight each)

OPERATION

Procedure Detail:
1. Make sure the funnel gate is closed.
2. Pour the sample to the funnel.
3. Make sure the containers are well placed under each opening.
4. Open the funnel gate, make sure the sample flow smoothly to the containers (hold the containers if needed)

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:
1. Print the form for divider QC that is available at \\Grainbin\QCdata\Equipment\dividers.
2. Record the date of test on equipment check interval located near the oven moisture.
3. Do the dividers monthly check using determined corn sample that is available in the lab.
4. Weight the sample to get 1000g, do the test on dividers, and follow the operation instruction.
5. Weight every container using Mettler SB-16000.
6. Record the date of test and test results in the form and put in the LAB QC book.
7. Record the data in the spreadsheet at \\Grainbin\QCdata\Equipment\testwt\dividers\boerner.
8. Calculate the weight percentage, average of weight percentage, differences from known measurement and average of difference percentage of every weight portion by copying the formula from previous data/measurement.
9. Update the UCL, mean, and LCL by copying the formula from previous data/measurement.

10. Update the control chart by dragging the deviation percentage data, UCL, mean and LCL. Sometimes the chart can update automatically if all the series has been dragged to the many empty lines before.
11. Print the updated chart and put in the LAB QC book.
12. Make the quarterly report, the format of report is available at \\Grainbin\QCdata\Equipment QC\Equipment Check Report.

If the sample is changed:

1. Check the previous sample control charts; make sure they have the right range of row.
2. Type new sample number with yellow highlight, continue to collect data, follow the previous sample example.
3. Build new control charts for new sample.

THE USE OF DATA

The divider monthly check data are used for the quality control purposes. The accuracy of divider will affect the activities done in the lab such us division of sample into small portion. The error can be prevented before the activity goes further.

Evaluation and Monitoring:

- Do quality control and maintenance monthly.
- Analyze the control chart.
- The test result should fall within the control limit.
- The out of control data should not exceed 5% of the total data.
- Print the control charts quarterly.
- Update the report quarterly.

Corrective Actions:

- Redo the measurement when the results fall outside the control limit.
- Check the balance that is used.
- When the balance is in good condition and the results are still out of tolerance (more than 5% of the data), consult with Lab manager to determine what action should be taken.

Grain Quality Laboratory
Iowa State University

PROCEDURE: Rotary Grain Divider

Background:
Sometimes only a part of the sample needed during a lab procedure. The grain divider divides the sample into smaller portions. (10%, 20%, 30% and 40%)

Objective:
To equally divide the sample into smaller portions. (10%, 20%, 30% and 40% of weight)

OPERATION

Procedure Detail:
1. Turn the machine on, make sure the funnel gate is closed.
2. Pour the sample to the funnel.
3. Make sure the containers are well placed under each opening.
4. Open the funnel gate.
5. Turn the machine off when you are done.

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:

13. Print the form for divider QC that is available at \\Grainbin\QCdata\Equipment\dividers.
14. Record the date of test on equipment check interval located near the oven moisture.
15. Do the dividers monthly check using determined corn sample that is available in the lab.
16. Weight the sample to get 1000g, do the test on dividers, and follow the operation instruction.
17. Weight every portion of weight (10%, 20%, 30%, 40%) using Mettler SB-16000.
18. Record the date of test and test results in the form and put in the LAB QC book.
19. Record the data in the spreadsheet at \\Grainbin\QCdata\Equipment\testwt\dividers\rotary.
20. Calculate the weight percentage, average of weight percentage, differences from known measurement and average of difference percentage of every weight portion by copying the formula from previous data/measurement.
21. Update the UCL, mean, and LCL by copying the formula from previous data/measurement.
22. Update the control chart by dragging the deviation percentage data, UCL, mean and LCL. Sometimes the chart can update automatically if all the series has been dragged to the many empty lines before.

23. Print the updated chart and put in the LAB QC book.
24. Make the quarterly report, the format of report is available at \\Grainbin\QCdata\Equipment QC\Equipment Check Report.

If the sample is changed:

4. Check the previous sample control charts; make sure they have the right range of row.
5. Type new sample number with yellow highlight, continue to collect data, follow the previous sample example.
6. Build a new control charts for the new sample.

THE USE OF DATA

The divider monthly check data are used for the quality control purposes. The accuracy of divider will affect the activities done in the lab such us division of sample into small portion. The error can be prevented before the activity goes further.

Evaluation and Monitoring:

- Do quality control and maintenance monthly.
- Analyze the control chart.
- The test result should fall within the control limit.
- The out of control data should not exceed 5% of the total data.
- Print the control charts quarterly.
- Update the report quarterly.

Corrective Actions:

- Redo the measurement when the results fall outside the control limit.
- Check the balance that is used.
- When the balance is in good condition and the results are still out of tolerance (more than 5% of the data), consult with Lab manager to determine what action should be taken.

Grain Quality Laboratory
Iowa State University

PROCEDURE: Seed Counter

Background:
Seed weight (usually expressed as grams per 1000 seeds or seeds per pound) is sometimes an important factor for food use grains. The seed counter passes seeds singly through a photoelectric counting beam; the weight of counted seeds allows computation of seed weight. Moisture content must be known because the weight of water in a seed will influence its 100 seed weight.

Objective:
To determine the weight of individual seeds expressed as gr/1000 seeds or seeds/lb.

OPERATION

Procedure Detail:
1. Turn on the power switch located on the counting box.
2. Press the red button on display panel to zero counting.
3. Place a receptacle (beaker or some other container) beneath the chute.
4. Load grain into the counting bowl.
5. Turn on the power switch located on the counting bowl.
6. Verify counter response as the grain climbs the bowl and passes the sensor.
7. Use a spatula to remove non-seed materials (do not count them!).
8. Turn off the power switch located on the counting bowl when the seed count reaches the predetermined number (usually 200) or when there are no seeds remaining in the counting bowl (for yearly and weekly quality control and maintenance)
9. Record number of seeds in the QC books and spreadsheet
10. Turn off the power switch located on the counting box and place all predetermined seeds in the drawer

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:
1. Print the seed counter check form which is available at \\Grainbin\QCdata\Equipment\Seed Counter.
2. Record the date of test on equipment check interval located near the oven moisture.
3. Do the yearly test on all the corn and soybeans seed (100, 200,300, 400 and 500 seeds)
4. Do weekly test on 300 seeds of corn and soybeans.
5. Record the date of test and test results in the form and put in the LAB QC book.

6. Record the data in the spreadsheet at \\Grainbin\QCdata\Equipment\Seed Counter.
7. Update the control limit by copying the formula from previous data/measurement.
8. Update the control chart for corn and soybean by dragging the data, UCL, mean and LCL. Sometimes the chart can update automatically if the all the series has been dragged to the many empty lines before.
9. Print the updated chart and put in the LAB QC book and post in the lab (near the seed counter).
10. Make the quarterly report, the format of report is available at \\Grainbin\QCdata\Equipment QC\Equipment Check Report

THE USE OF DATA

The seed counter data as a part of quality control procedure will be used for the NIRS calibration purposes. The data may affect the accuracy of NIRS calibration. The periodic check is done to make sure the device is ready to use and accurate.

Evaluation and Monitoring:

- Weekly check when counter is being operated - run precounted 300 seed reference.
- Annually check – recount 100, 200, 300, 400, 500 seed reference samples.
- Follow Scale Check SOP for maintenance of scales used for seed weight.
- Analyze the weekly data whether they exceed the control limit.
- The out of control data should not exceed 5% of the total data.
- Print the control charts and place it in the Lab QC book and near seed counter (quarterly).
- Update the report quarterly.

Corrective Actions:

- If errors happen in running weekly or annually check, rerun. If still in error, recount reference, consult operators manual. Record results in Operating Record for seed counter.
- When the weekly results exceed 5% of the total data, consult with the lab manager to take appropriate actions (seed counter may need to be repaired).

Grain Quality Laboratory
Iowa State University

PROCEDURE: Test Weight (Grain)

Background:
Grain Quality Lab needs to do the test weight for the purpose of quality control maintenance, in order to support research and service. The test weight is done using both the balance and GAC instrument.

Objective:
To find the weight of samples in a known volume in pound per bushels.

OPERATION

Procedure Detail:
1. Make sure the funnel is closed, the kettle is well and evenly placed and the catch pan is located under the kettle.
2. Take the grain (corn) sample that is available in the lab.
3. Pour the sample into the funnel.
4. Open the funnel to let grain flow into the kettle. There should be enough grain to overflow. If the sample is not large enough to fill any of the kettles do not proceed with the test.
5. Level the kettle with one W-motion stroke.
6. Make sure the balance (Seedburo 8800) has a pan on it and is at zero. If not press zero. Pour the sample in the pan.
7. Press [lb/bu] to convert the units from grams to lb/bu.
8. If you had used the quart kettle, record the number as it appears on the balance. If you had used the pint kettle, multiply the number by 2 and record the results.
9. Do step 1-8 using both quart and pint kettle.
10. Do the test weight using GAC 2000 and GAC 2100.
11. Follow the instructions on how to use the GAC instruments that are available near the instruments.

QUALITY CONTROL AND DATA HANDLING

Procedure Details:
1. Print the test weight form which is available at \\Grainbin\QCdata\Equipment\testwt.
2. Record the date of test on equipment check interval located near the oven moisture.
3. Do the grain test weight on kettle and GAC 2000/2100 weekly.

4. Record the date of test and test results in the form and put it in the LAB QC book.
5. Record the data in the spreadsheet at \\Grainbin\QCdata\Equipment\testwt.
6. Calculate the average and standard deviation of the test weight (for quart, pint, GAC 2000, GAC 2100) and moisture by copying the formula from previous data/measurement.
7. Calculate the different between Quart and Pint, and between GAC 2000 and GAC 2100 by copying from the previous data.
8. Calculate the difference between average of quart and pint, and average of GAC 2000 and GAC 2100, update the control limit for this different by copying from previous data.
9. Update each chart (Quart/Pint, GAC 2000/2100 test weight, GAC 2000/2100 moisture, difference between Quart/Pint and GAC 2000/2100) by dragging the data for each measurement item. Sometimes the chart can update automatically if the all the series has been dragged to the many empty lines before.
10. If the sample is changed:
    - Update the last line of average, standard deviation of the old sample.
    - Update the last line in the all the charts, make sure they are right.
    - Type the new sample number and highlight it using yellow color.
    - Copy the format and formula from the previous sample.
    - Build the new charts by copying from previous sample and change their start line/row.
11. Print the updated chart and put in the LAB QC book and post in the lab (near the Seedburro 8000 and GAC 2000/2100).
12. Make the quarterly report, the format of report is available at \\Grainbin\QCdata\Equipment QC\Equipment Check Report

THE USE OF DATA

The test weight data will be used to make sure that the instruments (GAC 2000/2100) and the balance are working accurately. When the undesirable data appear, actions to prevent the error can be determined.

Evaluation and Monitoring:

- Do the quality control and maintenance weekly.
- Compare the test weight result between quart/pint kettle and GAC 2000/2100.
- Analyze the chart to determine whether they are acceptable or not.
- The out of control data of difference between average of Quart/Pint and average of GAC 2000/2100 should not exceed 5% of the total data
- Change the grain sample yearly.
- Print the charts quarterly.
- Update the report quarterly.

Corrective Actions:

- Redo the measurement when the result is undesirable.
- Make sure to do procedure test weight cup correctly.
- Check the balance whether it is really in correct place.
- For GAC 2000/2100, make sure to choose the correct grain selection.
- When the measurement still show the undesirable result, consult with Lab manager, discuss it in the weekly meeting to determine what action should be taken.

Grain Quality Laboratory
Iowa State University

PROCEDURE: Test Weight (Water)

Background:
Grain Quality Lab needs to do the water test of test weight for the purpose of quality control maintenance. The accuracy of water test weight will determine the accuracy of test weight done in the lab.

Objective:
To find the weight of water for one quart and one pint in grams.
1 quart of water = 1098.1 g
1 pint of water = 549.05g

OPERATION

Procedure Detail:
1. Boil approximately 1.5-2.0 qts of water and cool at room temperature.
2. Pour the water into quart kettle carefully until it is full, try to not spill the water.
3. Tare the balance until it shows zero.
4. Weight the water using balance Mettler PM-4000 or Seedburro 8000, do this carefully and try to not spill water because this will affect the result.
5. Repeat step 2-4 for pint kettle.

QUALITY CONTROL AND DATA HANDLING

Procedure Detail:
1. Print the water test weight form which is available at \\Grainbin\QCdata\Equipment\testwt.
2. Record the date of test on equipment check interval located near the oven moisture.
3. Do the yearly water test weight, follow the operation procedure.
4. Do the test three times in each kettle.
5. Record the date of test and test result in the form and put it in the LAB QC book.
6. Record the data in the spreadsheet at \\Grainbin\QCdata\Equipment\testwt\water.
7. Calculate the average deviation from known measurement by copying the formula from previous data/measurement.
8. Update the chart by dragging the deviation data for both quart and pint measurement. Sometimes the chart can update automatically if the all the series has been dragged to the many empty lines before.

9. Print the updated chart and put in the LAB QC book.
10. Summarize the average of measurements, standard deviation, CV, difference between known and measured data for all the years.
11. Make the quarterly report (if the data is available), the format of report is available at \\Grainbin\QCdata\Equipment QC\Equipment Check Report.

THE USE OF DATA

The water test weight data are collected for the quality control purposes. This data may affect the accuracy of activity done in the lab. The error may be prevented before such activity goes further.

Evaluation and Monitoring:

- Do quality control and maintenance yearly.
- Analyze the data and chart.
- The deviation between known and measured data should be within the tolerance values (±1 gram)
- Print the chart and update report yearly

Corrective Actions:

- Redo the measurement the difference between known and measured data are not within the tolerance limits.
- Make sure to do procedure correctly, check the balance.
- If the errors still happen, consult with Lab manager to determine what action should be taken.

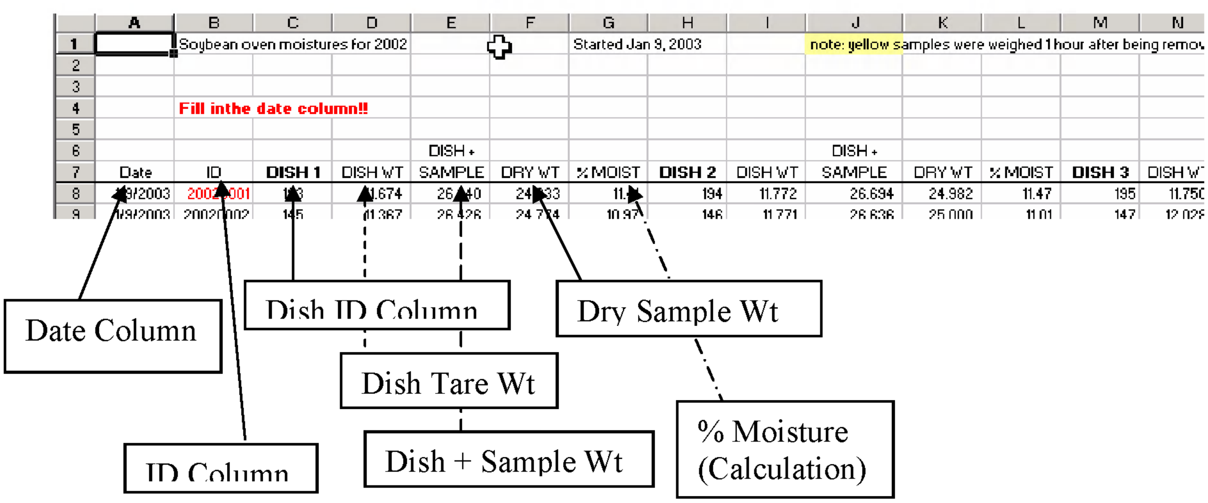
Grain Quality Laboratory
Iowa State University

PROCEDURE: Oven Moisture Measurement

Background:
This procedure is to determine the moisture value of corn and soybeans samples.

Objective:
To accurately measure whole grain moisture that will match the official moisture ovens at GIPSA\FGIS using the official methods (corn AACC 44-15A, soybeans AOCS Ac 2-41).

Procedure Detail:
1. Open the Spreadsheet oven moisture template found at
   \\Grainbin\references\soybeans\ovenmoisture\soybean oven moisture template.xls -or-
   \\Grainbin\references\corn\ovenmoisture\corn oven moisture template.xls
2. Make sure the information at the top of the spreadsheet is filled in correctly.
3. Use the 'save as' command to save the spreadsheet with the correct name, ie corn is cornXXXX where X is the current year.
4. Make sure the sample is well mixed.
5. Fill in the date and ID columns as seen below. (A8 and B8).
6. Locate the tray of drying dishes and enter the lowest ID number in the Dish 1 column (C8). The nest two dish ID's will be entered automatically.



7. Record the weight of the first dish using the balance next to the computer at the weigh station. To record the weight have the cursor cell on the correct cell and press F8, the weight will automatically be recorded.

8. Add the required amount of sample to the dish and record the weight in the Dish plus Sample column.
9. Repeat steps 7-8 until the three dishes are filled for one sample.
10. Place the samples in the oven when the oven is up to temperature.
11. Remove the samples after the method time has expired.
12. Place the samples in the desecrator until the sample temperature has equilibrated to room temperature.
13. Remove sample tray from the desecrator and find where it is located on the spreadsheet. Make sure the dish number matches the one on spreadsheet. Place the cursor cell on the Dry weight cell and press F8.
14. Dump the sample in the bucket after you are done.
15. Go to the next sample and repeat steps 13-14 until you are done.

QUALITY CONTROL AND DATA HANDLING

1. Build chart of average moistures vs. averages differences of three sample replicates for dishes.
2. Build chart of average moistures vs. averages differences of three sample replicates for duplicates.
3. Build chart of consecutive number vs. standard deviation for dishes and duplicates
4. Build 95% confidence level of differences between dishes and duplicates.
5. Move the average moisture and standard deviation of dishes and duplicate to Access Database.

THE USE OF DATA

The quality control data is used to evaluate the performance of oven moisture, to decide whether operator training is needed and to improve oven moisture data management possibility.

Evaluation and Monitoring:

- Pay attention to average differences of each pair of replicate for the dishes and duplicates**.** The tolerances for these differences follow GIPSA/USDA Method below:
  CORN: $\leq$ 0.25%, for $\leq$ 15% moisture
  $\leq$ 0.30%, for > 15% moisture
  SOYBEANS: $\leq$ 0.25%, for all moistures
  Calculate the out of control data, the formula to do this is available in the template spreadsheet. In the end, out of control should not exceed 5%.
- The out of control data of differences between dishes and duplicates should not exceed 5%.
- Evaluate the standard deviation chart whether it is acceptable.

Corrective Actions:

- If the wrong actions happen during the operation of oven moisture, contact Lab Manager.
- Always pay attention to the data collection, make sure the sample number is right.
- Discuss the quality control data to decide what action should be taken for the better performance.

Grain Quality Laboratory
Iowa State University

PROCEDURE: AccuPyc 1330 Pycnometer

Background:
The Pycnometer is an instrument to measure true densities in materials. Density is measured in units of grams per cubic centimeters (g/cc). The Grain Quality Laboratory uses these densities as a reference value for our corn density calibration.

Objective:
To obtain accurate density results of corn and soybeans

Procedure Detail:

START-UP

1. Flip on the power switch located at the upper right rear of the instrument just above the power cord.
2. **ALLOW TWO (2) HOURS FOR WARM-UP.**
3. Turn on the data storage device (printer or computer depending on specific instructions from lab supervisor).
4. Open the main gas valve on the top of the nitrogen tank.
5. Run the **density standards** following the analyze steps below.
6. Check the results against their values and tolerances posted. If there is any deviation, contact the lab supervisor.

ANALYZE

1. Weigh a sample into the tarred sample cup and record the weight on data sheets.
2. Place the cup with the sample into the pycnometer.
3. Close the chamber cap with a clockwise motion.
4. Press the white button in the upper right of the keypad, a plus (+) sign will appear in the upper right corner of the display.
5. Press the four (4) button, which is labeled analyze.
6. Enter the sample ID using the numbered section of the keypad and press the enter button.
7. Enter the sample weight (in grams) the was taken in step 1 and press the enter button.
8. Press the enter button again to start the analysis.
9. While machine is working, run the sample through the GAC 2000 and GAC 2100 to gather the moisture and density from the machines and enter into the template for this job.
10. When analysis is complete, press the CHOICE button to view results on the display.
11. Enter the results in the data sheet determined by the lab supervisor.
12. Open the chamber cap with a counter-clockwise motion.
13. Remove the sample and return it to its proper container.

14. Repeat steps one (1) to thirteen (13) until all samples have been analyzed.

NOTES

- When the white button is pressed and a plus sign appears in the upper right corner of the display (ANALYZE Step 4), the white options above the number keys are available for use.
- The FILL, EXPAND, and VENT options are accessible in the manual mode. To access the manual mode, press the white button and then the one (1) button.
- The escape (CLEAR) button can be used to stop or cancel any operation.
  An asterisk (*) in the upper right corner of the display is to notify the operator of error messages. To read the error messages press the CHOICE key to step through the messages and then notify the lab supervisor.

DATA COLLECTION

Corn density data collection is done during yearly validation/calibration NIR instruments.
Procedure Details:
1. Open Corn Density template at \\Grainbin\reference\Corn\Density\Cndenstemplate.
2. Use Pyconometer to measure density of corn samples.
3. Follow the procedures how to use Pyconometer as describe in "ANALYZE" section.
4. Do measurements three times, record each result to the spreadsheet.
5. While analyzing density measurements, do moisture and test weight measurements using GAC 2000 and GAC 2100 (three times each).
6. Record the results in the spreadsheet.
7. Calculate the average of density, moisture and test weight.
8. Convert the average density of three times measurement to the 15% moisture basis (moisture basis use average of six times measurements)
9. Build the chart of standard deviation of density (3 times measurements) and moisture (6 times measurements)
10. When data collection is done, move the 15% density to the Access DB.

QUALITY CONTROL AND DATA HANDLING

Quality control activity is performed daily during the density measurement of yearly calibration samples.
Procedure Details:
1. Use corn sample available near pyconometer (ask Lab manager).
2. Use sheet for daily check.
3. Name the sheet with the corn sample number.
4. Follow the procedure in the "Data Collection" section.
5. Build the 95% confidence level control chart for 15% density.

THE USE OF DATA

The quality control data is used to evaluate the performance of pyconometer and overall corn density activity, to decide whether operator training is needed and to improve corn density data management possibility.

Evaluation and Monitoring:

- Analyze the standard deviation chart of density and moisture
- Analyze the control chart from pyconometer daily check; the out of control data should not exceed 5%.

Corrective Actions:

- If the wrong actions happen during data collection, consult with Lab Manager
- When the out of control data exceed 5%, discuss it in the Lab meeting to decide what action should be taken.

Grain Quality Laboratory
Iowa State University

PROCEDURE: Reference Chemistry Databases

Background:
- Old reference chemistry databases were not well organized.
- The reference chemistry databases need to be well documented to support quality control program.
- "Sloppy records and poor records maintenance likely reflect poor quality control in other areas of operations and will give that impression to others". (Garfield, 1991).

Objective:
To develop well organized reference chemistry databases as part of quality control program.

Procedure:
STEP TO BUILD NEW DATABASES:
1. The old reference chemistry databases are organized and separated according to the table that will be built. (in Excel)
2. Additional information are added to the new tables, such as date of the test and Lab doing the test.
3. The individual files such as calibration, oven moisture, density might be useful to find information about the samples.
4. The new databases in the Excel are imported to the Access.

STEP TO LINK TABLES:
1. Save Sheet in Excel in "webpage format" (This will be table in Access).
2. Open Access, Click Get External Data- Link Table.
3. When the dialog box appears, choose "html document" for the file type.
4. Select table that you want to connect.
5. Follow step in the dialog box, before you finish, click "advanced" to make sure the field formats are right.

STEP TO ADD NEW DATA (UPDATE) TO THE DATABASES:
1. Find the respective files for each table, for example:
   - To update Master table, use the calibration file for the respective year.
   - To update Proximate table, use the Proximate Results from Eurofins (In the Completed form Folder) for the respective year.
   - See the "Reference Chemistry Files Location" Table
2. Copy the files according to the field to the Excel Databases, you may need to arrange the file before making a copy to Excel Databases.

3. Save the file as the table name in the "webpage" format.
   For example: If you update Proximate table, save "Proximate" sheet in Excel as webpage format, choose "selection sheet", save as "Proximate", it will replace the existed file.
4. Open Access DB, import updated file (webpage format/html documents)

STEP TO IMPORT FILE:

1. Click File-Get external data-import.
2. Click File type: html documents, choose the right file, click import.
3. Check box: First row contain column heading.
4. Click advanced, fill the data types of each field (see Data Type table)
5. Click next, check box "in a New Table"
6. Click next, check box "no primary key"
7. Click next, name the table, click finish.

STEP TO QUERY:

1. Click on "Queries", choose "Create Query in Design Views"
2. Choose table needed.
3. Connect ID3 of each table.
4. Choose Fields needed, fill the criteria, for example: >20040000 (ID3) if you want sample number greater than 20040000.
5. Click run (!)

STEP TO PERFORM CALCULATION:

1. Click on "Queries", choose "Create Query in Design Views"
2. Make query with fields needed from tables.
3. Type formula in the field design view, using right click "zoom"
   Ex. Total: [Protein 13%] + [Oil 13%]
3. Click run (!)

STEP TO USE AVERAGE OF REPLICATIONS (if samples have some measurements)

1. Click on "Queries", choose "Create Query in Design Views"
2. Make query with fields needed from tables.
3. Click on query design view
4. Choose ID3 to be in query.
5. Right click in the ID3 design view, click on "total" (Total will appear in design view fields)
6. Right click on "total" , choose "group by"
7. In the field that you want to calculate average of replication (blank field), choose "avg" and type for example, Ave Protein: Protein 13%
8. Click run (!)

Grain Quality Laboratory
Iowa State University

PROCEDURE: Process for Receiving and Storing Outside Lab Data

Background:
ISU Grain Quality Lab obtains reference chemistry data from external laboratory. Procedure of receiving and storing external data is needed to organize external data.

Objective:
To keep the external reference data well organized and easy to be searched.

Procedure Details:

1. Samples are chosen for lab analysis.
   Note: Presently Eurofins, University of Missouri, ISU\Fehr

2. A chemistry work order is completed.
   Filename protocol; GG LLL xx-xx-xxxx.xls, G=grain, L=lab, xx-xx-xxxx = date
   Stored in gb/ref/chemistry data from labs/grain/
   Paper copy in black notebook (annual record). Kept in GR office.
   Three tabs in work order – order, ids (if needed), results.
3. Samples are prepared. Work order count, ids matched against actual. Initial work order
4. Samples are shipped. Date on work order.
5. Data returns either (or both) in paper or electronic form.
   Electronic data is pasted into third tab (results) on work order. Original file saved in original data directory (Data Files From Labs)
   Paper data is entered using template pasted into third tab of work order.
   Analysis, compilation of duplicates and reproducibility checks is done immediately on receipt of data, in same spreadsheet tab.
   Work order (electronic copy and GR paper copy if desired) is updated with required date information.
   Paper copies saved in annual record (see 2.), then transferred to long term archive with copy of work order.
6. Data is copied from storage worksheet as needed for use.
7. Duplicates and reproducibility checks are added to archive records monthly.

REFERENCES CITED

AACC International. 2000. Approved Methods of the American Association of Cereal Chemist, 10th Ed. Method 39-00. The Association: St. Paul, MN.

AACC International. 2000. Approved Methods of the American Association of Cereal Chemist, 10th Ed. Method 78-60. The Association: St. Paul, MN.

AOAC International. 2002. *AOAC Training Courses Series: Measurement Uncertainty in the Testing Laboratory*. AOAC International, Gaitsburg, Maryland, USA.

AOCS International. 2006. *AOCS Laboratory Proficiency Program.* Available at: http://www.aocs.org/tech/lpp.asp. Accessed 22 November 2006.

Dorsey-Redding, C., C.R. Hurburgh, Jr., L.A. Johnson, and S.R. Fox. 1991. *Relationships Among Maize Quality Factors.* Cereal Chemistry. 68(6): 602-605.

Dux, James P. 1990. *Handbook of Quality Assurance for Analytical Chemistry Laboratory*. Van Nostrand, New York.

Garfield ,Fredrick M. 1991. *Quality Assurance Principles for Analytical Laboratories*. AOAC International, Gaithersburgh, MD, USA

Garfield ,Fredrick M, Eugene Klesta, Jerry Hirsch. 2000. *Quality Assurance Principles for Analytical Laboratories.* AOAC International, Gaithersburgh, MD, USA

Gryna, Frank M. 1999. *Quality and Costs*. Juran's Quality Handbook, Ed by Joseph M. Juran and A. Blanton Godfrey. McGraw-Hill, New York, USA.

Hansen, Audrey M. 2004. *Establishing a Quality Management System in a University-Based Analytical Laboratory*. Master Thesis, Iowa State University, Ames, IA.

Hruschka, William R. 2001. *Near-Infrared Technology in the Agricultural and Food Industries*, Data Analysis: Wavelength Selection Method. Edited by P.C. Williams and Karl Norris. American Association of Cereal Chemist, Minnesota, USA.

Hurburgh, C.R. Jr. 2003. *Quality Management Systems for Agriculture*. Engineering and Technology for a Sustainable World. April: 13-16.

Iowa State University Extension. 1998. *Iowa Gold Catalog High-Oil Corn Test*. Iowa State University Extension, Ames, IA.

Kateman G. and L. Buydens. 1993. *Quality Control in Analytical Chemistry*. Edited by J.D. Winefordner and I.M. Kolthoff. John Wiley and Sons, Inc, New York.

Marsilli, Ray. 1997. *Sending It Out: Outsourcing Pros and Cons. Food Product Design. Weeks Publishing Company*. Available at: http://www.foodproductdesign.com/archive/1997/0597QA.html. Accessed 9 August 2005.

Morris, Alan S. 1991. *Measurement and Calibration for Quality Assurance*. Prentice Hall International, UK.

Steel, Robert G.D, James H. Torrie and David A. Dickey. 1997. *Principles and Procedures of Statistics: A Biometrical Approach, 3rd Ed.* The Mcgraw-Hill Companies, Inc, USA.

Thakur Maitri. 2006. *Quality and Nutritional value of United States Soybeans and Soybeans Meal*. Master Thesis, Iowa State University, Ames, IA.

Tholen, D., *An Introduction to ISO 17025 and Laboratory Accreditation*. AOAC International Training Course Materials. Attended by A.M. Hansen, 6/12//2002.

Westgard, James O, Patricia L. Barry. 1986. *Cost Effective Quality Control: Managing the Quality and Productivity of Analytical Processes*. AACC Press, Washington DC.

Williams, P.C. and K.Norris. 2001. *Near-Infrared Technology in the Agricultural and Food Industries,* Variables Affecting Near-Infrared Spectroscopic Analysis. Edited by by P.C. Williams and Karl Norris. American Association of Cereal Chemist, Minnesota, USA.

Wilson,D. *1995. The Cost of Quality*. Educesoft. Available at: http://www.educesoft.com/quality/costofquality.htm Accessed 20 March 2006.

Workman, Jerome J. 1996. Jr. *Interpretive spectroscopy for near infrared.* Near Infrared Spectroscopy: The Future Waves, Ed by A.M.C Davies and Phil Williams. NIR Publications, Chicester, UK, p.6.

Vaderman, Stephen B and J.Marcus Jobe. 2001. *Introduction to Formal Statistical Inference*. Basic Engineering data Collection and Analysis. Duxbury, Thomson Learning, California, USA.

National Type Evaluation Program. 2004. *Type Evaluation Test Procedures and Tolerances Near-Infrared Grain Analyzers*. Grain Moisture Meters and Near Infrared Grain Analyzers: Near Infrared Grain Analyzer. NCWM Publication 14.

USDA-GIPSA. 1996. *USDA Handbook Chapter 5*. USDA-GIPSA, Washington DC, USA.

USDA-GIPSA. *1998. Moisture Reference Program: Air Oven Reference Method for Moisture in Corn and Beans.* USDA-GIPSA, Washington DC, USA.